United States Court of Appeals for the Second Circuit



APPENDIX

74--1050

United States Court of Appeals

For the Second Circuit.

THE GENERAL TIRE & RUBBER COMPANY,

Plaintiff-Appellant,

v.

JEFFERSON CHEMICAL COMPANY, Inc., Defendant-Appellee.

ON APPEAL FROM THE UNITED STATES DISTRICT COURT FOR THE SOUTHERN DISTRICT OF NEW YORK.

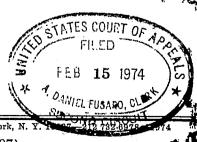
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PAGINATION AS IN ORIGINAL COPY

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6,	U. S. Patent No. 2,527,970, Herman Sokol	702a
7B.	Bayer, Otto "Industrial Application of the "Diisocyanates," Angewandte Che- mie, Vol. 59: 257-272 (Sept. 1947) (Excerpts, pages 1-31, 41)	707a
9,	U. S. Patent No. 2,726,219, Frederick B. Hill, Jr.	74()a
10.	"Princeton Introduces Novel Polyure- thanes Made From Castor Oil and Diisocyanate," Chemical Engineering, Vol. 57, No. 4, April 1950 pages 79, 165-166	74 2a
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779a	"Dow Introduces 5 Newcomers in Family of Polyglycols open new fields of applications," Chemical and Engineering News, page 71 (December 16, 1957)	15.
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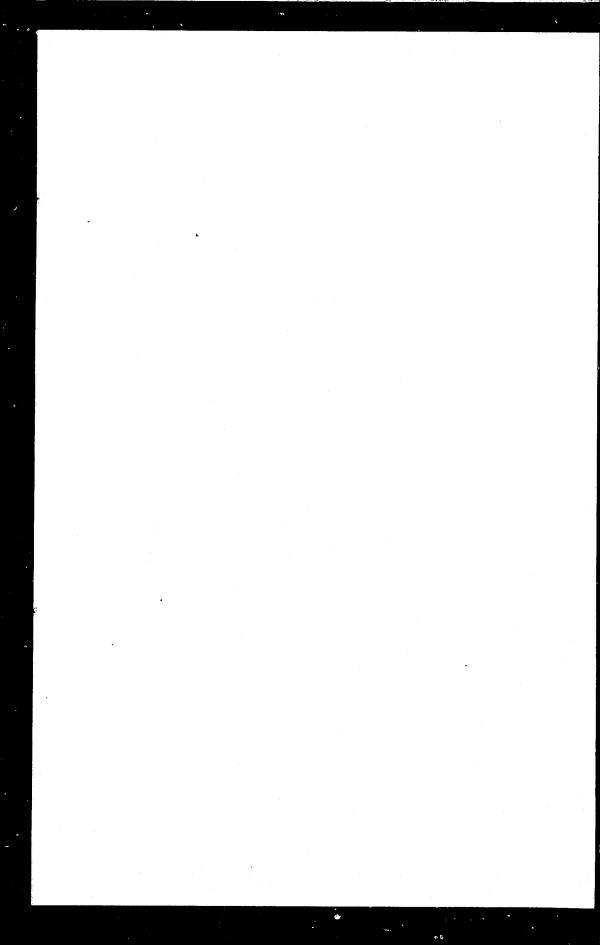
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United States Court of Appeals

FOR THE SECOND CIRCUIT.

Docket No. 74-1050

THE GENERAL TIRE & RUBBER COMPANY,

Plaintiff-Appellant,

v.

JEFFERSON CHEMICAL COMPANY, Inc.,

Defendant-Appellee.

On Appeal from the United States District Court for the Southern District of New York.

Relevant Docket Entries.

1968

Mar. 26 Summons and Complaint.

May 27 Defendant's Answer and Counterclaims.

June 18 Plaintiff's Reply to First Counterclaim.

1969

Apr. 1 Plaintiff's Reply to Second Counterclaim.

July 29 Plaintiff's motion to sever and transfer defendant's Second Counterclaim.

1970

May 6 Order Severing Defendant's Second Counterclaim and transferring same to District of Delaware.

Relevant Docket Entries

1971

Feb. 19 Stipulation and Order re amended pleadings.

Feb. 19 Amended Complaint.

Feb. 19 Amended Answer and Counterclaim.

Mar. 9 Amended Reply to Amended Counterclaim.

1972

Feb. 7 Note of Issue.

1973

Jan. 15-22 Trial before Judge Robert L. Carter.

Sept. 6 Opinion of Judge Carter.

Nov. 9 Judgment.

Dec. 6 Notice of Appeal.

UNITED STATES DISTRICT COURT.

SOUTHERN DISTRICT OF NEW YORK.

THE GENERAL TIRE & RUBBER COMPANY,

Plaintiff,

v.

JEFFERSON CHEMICAL COMPANY, Inc.,

Defendant.

Civil Action No. 68 1227

Plaintiff, for its complaint herein, avers:

- 1. Plaintiff, The General Tire & Rubber Company, is a corporation duly organized and existing under the laws of the State of Ohio and has its principal office and place of business at 1708 Englewood Avenue, Akron. Ohio.
- 2. Defendant, Jefferson Chemical Company, Inc., is a corporation organized and existing under the laws of the State of Delaware and is qualified to do business in the State of New York and maintains an office and does business at 260 Madison Avenue, New York, New York.
- 3. This is an action for a Declaratory Judgment under Title 28 U. S. C., Sections 2201 and 2202, in respect of an actual controversy of which this Court has jurisdiction under the Patent Laws of the United States, and under 28 U. S. C. Sections 1338 and 1391 (c).
- 4. Plaintiff is engaged, inter alia, in the business of manufacturing and selling polyurethane products and in-

tends to continue the manufacture and sale of such products.

- 5. Defendant claims to be the owner of U. S. Letters Patent No. 3,102,875 issued September 3, 1963 for "Polyurethane Reaction Product and Method of Making Same" on application Serial No. 373,036 filed August 7, 1953. Upon information and belief, ownership of said patent has been claimed by Mobay Chemical Company and the question of title to the said patent was the subject of litigation between defendant and Mobay Chemical Company in Civil Action No. 2950 in the Court of Chancery of the State of Delaware in and for New Castle County.
- 6. Defendant has charged plaintiff with infringement of United States Letters Patent No. 3,102,875.
- 7. Upon information and belief, and as presently advised, plaintiff admits that if the patent is valid, it has infringed claims 3, 4, 5, 6, 8, 9, 10 and 11 of said Letters Patent No. 3,102,875. Except as so admitted, plaintiff denies that it has infringed claims 1, 2, 7 and 12 of United States Letters Patent No. 3,102,875.
- 8. Upon information and belief, said claims 3, 4, 5, 6, 8, 9, 10 and 11 of United States Letters Patent No. 3,102,875 are each invalid and void for each of the following reasons:
 - (a) Because the alleged invention purported to be patented in and by said Letters Patent was, before the alleged invention thereof by the alleged inventor, Herbert L. Heiss, in all its material and substantial parts, known to and used by others in this country and patented or described in printed publications.
 - (b) Because more than one year prior to the filing of the application for Letters Patent No.

3,102,875, the alleged invention purported to be patented therein, was patented or described in printed publications in this or a foreign country and in public use and on sale in this country.

- (c) Because the alleged invention purported to be patented therein was described in patents granted on applications for patents by others filed in the United States before the alleged invention thereof by the said Heiss.
- (d) Because the said Heiss did not himself invent the subject matter sought to be patented therein.
- (e) Because the said Heiss was not the original, first, true or sole inventor of the alleged invention purported to be patented therein.
- (f) Because before the alleged invention thereof by the said Heiss, the alleged invention was made in this country by others who had not abandoned, suppressed or concealed the same.
- (g) Because the alleged invention purported to be patented therein did not constitute patentable invention within the meaning of the Patent Law in view of the prior state of the art in that any differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious, at the time the alleged invention was made, to a person having ordinary skill in the art to which said subject matter pertains.
- (h) Because the specification thereof does not contain a written description of the alleged invention, or of the manner or process of making and using it, in such full, clear, concise, and exact

terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same; and because the specification thereof does not set forth the best mode contemplated by the said Heiss of carrying out his alleged invention.

- (i) Because the claims of said patent do not particularly point out and distinctly claim the subject matter which the said Heiss regarded as his invention.
- (j) Because the alleged invention claimed in said patent is not the same as any invention disclosed in the application for patent filed August 7, 1953, and said claimed invention includes subject matter not adequately shown or described in said application.
- (k) Because the alleged invention purported to be patented therein, is broader than any alleged invention of said Heiss described in the specification of said patent and embraces compositions which in fact fail to accomplish the asserted objects or results described therefor and the alleged invention claimed in said patent is, therefore, inoperative and devoid of utility; and because the claims of the said patent are broader than the alleged invention described in the specification of said patent.
- 9. Upon information and belief, United States Letters Patent No. 3,102,875 is unenforceable because defendant has misused said patent.
- 10. Upon information and belief defendant is estopped to assert United States Letters Patent No. 3,102,875 against plaintiff by reason of laches.

11. An actual and justiciable controversy exists between plaintiff and defendant with respect to said patent and the alleged infringement thereof by plaintiff.

WHEREFORE, plaintiff prays this court to declare the rights and legal relations of the parties in respect of the controversy hereinabove set forth and to that end demands:

- T. That this court adjudge and decree that claims 3, 4, 5, 6, 8, 9, 10 and 11 of United States Letters Patent No. 3,102,875 are each invalid and void.
- II. That this court adjudge and decree that claims 1, 2, 7 and 12 of United States Letters Patent No. 3,102,875 have not been infringed by plaintiff.
- III. That this court adjudge and decree that plaintiff has the right to make, use and sell polyurethane products notwithstanding claims, 3, 4, 5, 6, 8, 9, 10 and 11 of United States Letters Patent No. 3,102,875.
- IV. That this court adjudge and decree that United States Letters Patent No. 3,102,875 is unenforceable.
- V. That this court issue a permanent injunction enjoining defendant, its officers, agents, employees, associates and attorneys, and those in privity with it, from representing to any person or persons whatsoever, excepting only in this court and in this action, that claims 3, 4, 5, 6, 8, 9, 10 and 11 of United States Letters Patent No. 3,102,875 are valid and from attempting to prevent plaintiff or its associates, affiliates or assignees, from freely making, using or selling the products which infringe said claims of United States Letters Patent No. 3,102,875.

VT. That plaintiff be awarded a reasonable attorneys' fee and the costs and disbursements of this action; and such other and further relief as to this Court may seem just and proper.

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Amended Answer and Counterclaim.

UNITED STATES DISTRICT COURT,

SOUTHERN DISTRICT OF NEW YORK.

[SAME TITLE.]

Amended Answer.

Now comes the Defendant, Jefferson Chemical Company, Inc., and for its answer to Plaintiff's Amended Complaint states as follows:

- 1. Defendant admits the allegations of paragraph 1 of the Amended Complaint.
- 2. Defendant admits the allegations of paragraph 2 of the Amended Complaint.
- 3. Defendant admits the allegations of paragraph 3 of the Amended Complaint.
- 4. Defendant admits that Plaintiff is engaged, interalia, in the business of manufacturing and selling polyurethane products and, to the best of Defendant's knowledge, that Plaintiff intends to continue the manufacture and sale of such products.
- 5. Defendant admits the allegation of paragraph 5 of the Amended Complaint and further states that it is the owner of U.S. Letters Patent No. 3,102,875. See Jefferson Chemical Company, Inc., v. Mobay Chemical Company, 167 U.S.P.Q. 108 (Del. Ch. 1970).
- 6. Defendant admits the allegations of paragraph 6 of the Amended Complaint.
- 7. Upon information and belief and as presently advised, defendant admits that plaintiff's infringement of United States Letters Patent No. 3,102,875 is limited to claims 3, 4, 5, 6, 8, 9, 10 and 11.

Amended Answer and Counterclaim

- 8. Defendant denies the allegations of paragraph 8 of the Amended Complaint and all of its sub-parts.
- 9. Defendant denies the allegations of paragraph 9 of the Amended Complaint.
- 10. Defendant denies the allegations of paragraph 10 of the Amended Complaint.
- 11. Defendant admits the allegations of paragraph 11 of the Amended Complaint.

Wherefore, Defendant prays this Court to

- I. Dismiss Plaintiff's Amended Complaint,
- II. Award Defendant reasonable attorneys' fees and costs,
- III. Award Defendant such other relief as to this Court may seem just and proper.

Amended Counterclaim.

Now comes the defendant, Jefferson Chemical Company, Inc., and for its amended counterclaim herein avers as follows:

- 1. Defendant, Jefferson Chemical Company, Inc., is a corporation organized and existing under the laws of the State of Delaware and has its principal office and place of business at 3336 Richmond Avenue, Houston, Texas.
- 2. Plaintiff, The General Tire & Rubber Company, is a corporation organized and existing under the laws of the State of Ohio, has its principal office and place of business at 1708 Englewood Avenue, Akron, Ohio, is licensed to do business within the State of New York and is doing business within the State of New York and this judicial district.

Amended Answer and Counterclaim

- 3. This counterclaim is for patent infringement, jurisdiction being based on Section 1338 of Title 28, United States Code.
- 4. Defendant is the owner of United States Letters Patent No. 3,102,875 entitled "Polyurethane Reaction Product and Method of Making Same" which was duly and legally issued on September 3, 1963.
- 5. Defendant is presently informed by Plaintiff and believes and, therefore, avers that Plaintiff, The General Tire & Rubber Company, has been and still is infringing claims 3, 4, 5, 6, 8, 9, 10 and 11 of said United States Letters Patent No. 3,102,875 by making, using and selling polyurethane foam embodying the patented invention of those claims and will continue to do so unless enjoined by this Court.

Wherefore, Defendant prays for a judgment granting a final injunction against further infringement of the said United States Letters Patent No. 3,102,875 by Plaintiff and those controlled by Plaintiff; an accounting; reasonable attorneys' fees; costs of this action; and such other and further relief as to this Court may seem just.

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Plaintiff's Amended Reply to Defendant's Amended Counterclaim.

UNITED STATES DISTRICT COURT,

SOUTHERN DISTRICT OF NEW YORK.

[SAME TITLE.]

Plaintiff, for its reply to defendant's Amended Counterclaim herein, avers as follows:

- 1. Plaintiff admits the averments contained in Paragraph 1 of defendant's Amended Counterclaim except that it has its principal office and place of business at One General Street, Akron, Ohio.
- 2. Plaintiff admits the averments contained in Paragraph 2 of defendant's Amended Counterclaim.
- 3. Plaintiff admits the averments contained in Paragraph 3 of defendant's Amended Counterclaim.
- 4. With respect to the averments contained in Paragraph 4 of defendant's Amended Counterclaim plaintiff admits that United States Letters Patent No. 3,102,875 entitled "Polyurethane Reaction Product and Method of Making Same" issued on September 3, 1963 but plaintiff denies that the said patent was duly and legally issued and as to the remaining averments of said paragraph plaintiff is without knowledge or information sufficient to form a belief.
- 5. With respect to the averments contained in Paragraph 5 of defendant's Amended Counterclaim, as presently advised, plaintiff admits, that if the patent is valid, plaintiff has been and still is infringing claims 3, 4, 5, 6, 8, 9, 10 and 11 of said United States Letters Patent No. 3,102,875 by making and selling polyurethane foam embodying the alleged invention of those claims and intends to continue to do so.

Plaintiff's Amended Reply to Defendant's Amended Counterclaim

As and for plaintiff's affirmative defenses to defendant's amended counterclaim:

6. Plaintiff repeats and reavers paragraphs 8, 9, and 10 of plaintiff's Amended Complaint as if here set forth in full.

Wherefore, plaintiff prays this Court to dismiss defendant's Amended Counterclaim and to award such other relief as to this Court may seem just and proper.

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By s/ Herbert Blecker

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UNITED STATES DISTRICT COURT,

SOUTHERN DISTRICT OF NEW YORK.

[SAME TITLE.]

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Kane, Dalsimer, Kane, Sullivan, Kurucz & Goldstein, Esqs., of New York City for Defendant; John Caslin, Esq., of Counsel.

Coffee & Sweeney, Esqs. of Chicago, Illinois for Defendant; James R. Sweeney, Esq., J. Robert Stapleton, Esq., Carl G. Ries, Esq., of Counsel.

Opinion.

CARTER, District Judge:

This action, brought on by plaintiff, General Tire & Rubber Co. (hereinafter called "General" or "plaintiff"), seeks judgment pursuant to 28 U.S.C. §§2201 and 2202 declaring certain claims in United States #3,102,875, issued on September 3, 1963, to Herbert Heiss (hereinafter called "The Heiss Patent"), now assigned to defendant, Jefferson Chemical Co. (hereinafter called "Jefferson" or "defendant"), invalid under 35 U.S.C. \$\$102(e), 102(g), 103 and 112. General alleges, in addition, that these claims are not the invention of Mr. Heiss: that they constitute late claims and should not be protected from the date the application itself was filed in the Patent Office but only from the date when the claims were added as amendments to the application; that the claims are invalid for double patenting and that the agreement

under which Jefferson acquired title to the patent violates public policy. Plaintiff concedes that if the validity of the patent is upheld, it has infringed the claims in question. Jefferson counterclaims for injunctive relief barring further infringement, for an accounting, costs and attorneys' fees. Jurisdiction exists over the subject matter and the parties pursuant to 28 U.S.C. §1400(b).

Background Facts:

Plaintiff is an Ohio corporation with its principal place of business in Akron, Ohio. It is engaged, *inter alia*, in the business of manufacturing and selling polyurethane foam products, and since 1964 has manufactured and sold over 100 million pounds of polyether urethane foam. Defendant, a Delaware corporation, maintains offices and is qualified to do business in New York.

The patent in controversy has been assigned to defendant pursuant to agreement between Mobay Chemical Corporation and defendant. This agreement provides that Jefferson use all reasonable efforts within not less than two years from the date of the agreement to secure at least one manufacturer of polyoxyalkylene polyether polyols as a non-exclusive licensee under The Heiss Palent pursuant to terms providing for royalty payments of not less than \$200,000 within a period of five years from the effective date of the license; moreover, defendant is required to make available licenses to additional polyoxyalkylene polyether polyol manufacturers on similar terms or on conditions requiring the payment of higher royalties: Jefferson is obligated to pay Mohav 40% of whatever funds it receives pursuant to such licensing and The Heiss Patent is to be reassigned to Mobay at the end of the two-vear period unless at least one license has been granted, or Jefferson has filed at least one complaint in a United States District Court charging at least one party with infringement.

Pursuant to the above agreement Jefferson, on January 25, 1968, having failed to obtain the license required, instituted an action against General in the Northern District of Illinois for infringement of The Heiss Patent (Civil Action 68 C 150). On March 23, 1968 General filed the instant action in this court, and thereafter the Illinois litigation was voluntarily dismissed.

The Heiss Patent:

The patent in question involves processes for making various polyurethane products such as adhesives, sealants, protective coatings and foams. The process for making foams by reacting a condensate of propylene oxide and a polyol such as pentaerythritol having three or four hydroxyl groups with an excess of diisocyanates, is the invention with which this controversy is concerned.

The File Wrapper History of the Prosecution of the Heiss Patent in the United States Futent Office:

The application which was to become The Heiss Patent had a very rough time progressing through the Patent Office. It was filed on August 7, 1953, in the name of James H. Saunders and Herbert L. Heiss as an invention of "Novel Resinous Compositions and Methods of Producing Same". It emerged some ten years later as U. S. Patent #3,102,875 under Heiss's name alone and as an invention of "Polyurethane Reaction Product and Method For Making Same." The first four pages of the initial application which was part of the file wrapper, admitted as an exhibit in this case, reads substantially the

Each of the parties has filed as exhibits copies of portions of the file wrapper in the prosecution of The Heiss Patent and the Price-Heiss interference. My assumption is that the portions furnished were those claimed by each party to be relevant to its contentions and that whatever has not been filed has no bearing on the issues at stake in this proceeding.

same as columns 1, 2 and through line 12 of column 3 of the patent as finally issued. In short, the invention described in the initial application as that of Saunders and Heiss bears, at least in part, the same description as the invention which, as patented, is attributed solely to Heiss. It should be added that Jefferson asserts in its post-trial brief that the specifications in the Saunders-Heiss application and those in the Heiss patent as issued are the same, and this assertion has not been controverted.

All claims in the initial application were rejected as unpatentable over various cited references² in a determination dated April 8, 1954,³ addressed to Saunders. New claims were then amended on October 7, 1954,⁴ and again all claims were rejected—this time in a communication to Saunders dated October 25, 1956, and in this communication a newly cited reference was named as rendering the application unpatentable.⁵

Further amendment was made on March 12, 1957, and once again all claims were rejected in a communication dated July 31, 1957. This determination contained newly cited references not mentioned in either of the prior re-

²Since the cited references are not claimed here as prior art, they will not be identified.

³The file wrapper containing action by the Patent Office usually bears a stamped date showing when the document was mailed from the Patent Office. This is the date usually referred to here as the date a determination was made by the Patent Office.

Where an amendment or other papers have been filed by the parties, I have sometimes used the date placed on the document by the patent attorneys, rather than the date stamped on by the Patent Office when the document was received. Neither in terms of chronology, clarification nor in any other respects do differences in these dates seem critical to this controversy.

Again, since the cited reference is not claimed here as prior art rendering the patent invalid, it has been omitted.

jections. A new amendment was filed on January 27. 1958, which was deemed an incomplete response. Further amendment was thereafter filed on February 17, 1958. On October 2, 1958, all claims were rejected as final over cited added references, including citations to Vol. 57 of Chemical Engineering. Further amendment was then filed in an effort to persuade the examiner to reconsider his final rejection.

Then, on December 10, 1958, patent #2,866,774 (hereinafter the "Price Patent") entitled "Polyether Polyurethane Rubber" was issued to Dr. Charles Price. learning of this patent a new amendment to the Saunders-Heiss application was filed on February 4, 1959, in which certain claims in the Price Patent were copied and made a part of the Saunders-Heiss application, and the examiner was requested to declare an interference between the Price Patent and the Saunders-Heiss application. On March 5, 1959, certain of these claims were rejected as unpatentable over newly cited references, but the examiner suggested a reformulation of certain of the claims and indicated if so reformulated, the claims might be allowed. The suggested reformulation was filed as an amendment on March 24, 1959, and an interference between Price and Heiss was declared on July 21, 1959.

In September 1959, Saunders and Heiss filed a petition with supporting affidavits requesting that their application be converted from Saunders-Heiss as joint inventors to Heiss as sole inventor, on the ground that the only remaining claims were those on which Heiss alone had worked. The conversion was granted on December 23. 1959.

⁶As I understand the procedure, Saunders and Heiss were asserting, by copying the Price claims, that the Price Patent and their application were to the same invention, and since their application had been filed before that of Dr. Price, that their application should be accorded priority; in effect, that the issuance of the Price Patent should be rescinded, and a patent issued to them.

Further amendment to the application followed on April 29, 1960, with the addition of four claims. On August 13, 1962, U. S. patent #2,948,691, issued to Erwin Windemuth, Herman Schnell and Otto Bayer (hereinafter referred to as the "Windemuth Patent") was cited as an additional reference, and the final rejection of October 2, 1958, was withdrawn. The allowed claims were rewritten as an amendment on September 17, 1962, and on October 12, 1962, certain of the claims were rejected and others allowed. At some point after Saunders' name was removed from the application, the title was changed and on September 3, 1963, the patent issued.

The Price-Heiss Interference:

Dr. Charles Price had filed his patent application on September 23, 1953, approximately six weeks subsequent to the filing date of the Saunders-Heiss application. The Price Patent calls for a polypropylene oxide condensate having a molecular weight of 600 or more to react with an excess of diisocyanates. The interference was allowed to go forward on the arguable thesis that Heiss' claims necessarily encompassed Price's claims because, while the former had placed no stress on molecular weight limitation, the polypropylene oxide condensate which was basic to the Price Patent and Heiss application inherently had a molecular weight limitation of 600 or more.

Dr. Price, in an affidavit filed in the course of the interference, gave January 1949, as his date of conception and April 1949, as the date of reduction to practice. Mr. Heiss gave November 14, 1951 as the date of con-

This Windemuth reference is the one on which plaintiffs rely as prior art rendering The Heiss Patent invalid to abviousness".

ception. Price was thus contending entitlement to priority over Heiss on the basis of prior conception and prior reduction to practice.

Dr. Price filed a motion to dissolve the interference, arguing that the polypropylene oxide condensate in question could vary all over the lot in respect of molecular weight, depending upon the process followed and reaction conditions, and that a molecular weight of 600 is not inherently obtained, citing Sokol, U. S. patent #2,527,970 (here urged as prior art), as a reference in support of this thesis. Moreover, Price argued, Heiss' process necessitated the presence of a plasticizer while his did not.

On August 15, 1960, the examiner denied the motion to dissolve on the ground that nothing in the disclosure in the Price patent showed that the condensate with a molecular weight limitation below 600 would not yield the same result as one with the molecular weight limitation his invention required. He also found that nothing in the specifications in the Heiss application necessitated the presence of a plasticizer and concluded that the language used made clear that the presence of a plasticizer was not critical. On October 3, 1960, in denying a motion to reconsider his denial of the motion to dissolve the interference, the examiner pointed out that he had taken cognizance of the Windemuth Patent, which, as indicated previously, is urged here as prior art.

The issue was taken to the Board of Patent Interference. There, as he had done before the examiner, Price stressed prior conception and reduction to practice and asserted that a molecular weight limitation of 600 was

The Heiss affidavit does not disclose the date of reduction to practice, but during the course of the interference and in these proceedings, he introduced testimony seeking to establish that date as September 19-22, 1952.

critical to his invention and that the polypropylene oxide condensate in question disclosed in both his patent and in the Heiss application would not inherently yield a molecular weight of 600.

The Board of Interference held that Heiss was in no way concerned with the molecular weight of the condensate required for his invention; that it was clear that the condensate disclosed in his application, when prepared in accord with the teachings of prior art, would be sufficient to meet his requirements without regard to molecular weight: and that Heiss claims had been triggered by knowledge of the Price Patent.9 On the other hand, the Board concluded that the Price patent established that the condensate there disclosed must have a molecular weight of at least 600 in order for his invention to be successfully practiced. It held that while the Heiss claims were subject to no such limitations and were broader than the claims made by Price, "the fact that a broad claim embraces the subject matter of a narrower one does not, in itself, establish that the two are drawn to the same invention," citing Andrews v. Wickenden, 194 F. 2d 729 (C. C. P. A. 1952), and Emerson v. Beach, 215 F. 2d 290 (C. C. P. A. 1954) (italics supplied). The Board decided that the Price claims with the molecular weight limitation, therefore, "are not for the same invention," and priority of invention in the subject matter in issue in the interference was awarded to Price (italies supplied). The Board did not deal with

The read this as referring to Heiss' emphasis on molecular weight of the condensate as being inherently 600 or more. The Board could mean no more than that since, as I understand it, in an interference the claims of one party are literally copied by the party seeking the interference to show that his claims and those of the other party go to the same invention.

any of the other questions raised. No appeal was taken by Heiss from that decision. 11

The Issue of Prior Art:

Plaintiff relies chiefly on the Windemuth Patent as prior art rendering The Heiss Patent invalid; in the alternative, it contends that Windemuth, an article by Dr. Otto Bayer, published in this country in 1947, U. S. patent #2,527,970 issued to Herman Sokol in 1960, and the Hill Patent, #2,726,219, issued in 1955, collectively constitute prior art rendering The Heiss Patent invalid for obviousness. Defendant contends that none of these teachings taken together or separately constitute prior art to The Heiss Patent. For reasons to be discussed below, I reject the arguments of both parties.

Dr. Bayer was, I gather, if not the father, one of the earlier inventive geniuses of the polyurethane industry which had its beginnings in Germany in 1937 with the development of polyester based urethanes. Bayer's great contribution was the teaching of a method of cross link-

¹⁰It should also be mentioned that the Price Patent cites Hill, U.S. Patent #2.726.219 (Hill Patent) as a reference, on which plaintiff also relies here as prior art.

the prosecution of the Heiss Patent and of the Price-Heiss interference at some length. Varying degrees of weight are attached by the courts to the action of the Patent Office. In this proceeding, I must determine de novo. independent of the Patent Office action, the validity of the patent in question, but where, as here, some of the same contentions are being made and issues raised which were raised and decided in proceedings in the Patent Office, Judge Mansfield's admonition in The Ansul Company v. Univoyal, Inc., 301 F. Supp. 273, 280 (S.D.N.Y. 1969). modified on other grounds, 448 F. 2d 872 (2d Cir. 1971), cert. denied, 404 U. S. 1018 (1972) that "it is unrealistic to attach any great weight to the allowance of a patent by an overworked staff [in the Patent Office]" seems wholly inapposite.

ing by use of diisocyanates thereby rendering polyester based urethane products more durable. Foams and adhesives were made by this process. The problem was that polyester urethanes were subject to destruction by hydrolysis. Moreover, diisocyanates, which were the most expensive ingredient, were required in large quantities and therefore the finished product was too costly for extensive commercial exploitation.

Working to solve this problem culminated in the Windemuth Patent, attributed to Dr. Bayer, Erwin Windemuth and Herman Schnell. This patent was issued in Germany in 1951, filed in this country on May 6, 1952,¹² and issued here on August 9, 1960. Its great advance over prior teaching was in the making of polyether (as opposed to polyester) based urethanes. Ethers are sensitive to hydrolysis but are not destroyed by water, as are esters. Moreover, here the diisocyanate ingredient necessary for

¹² The date of conception of the Heiss process is given as September 14, 1951 and the date of reduction to practice is given as September 1952 for the process involved here. In addition, defendant has produced notebook references which show that Heiss had the conception disclosed by Windemuth by October 10, 1951 and had reduced the Windemuth teaching to practice (making foams by reacting a condensate of ethylene oxide and glycerine with an excess of diisocyanates) by late October, 1951. The Windemuth Patent was filed in this country on May 6, 1952. Under these circumstances it is argued that Windemuth cannot be regarded as prior art to Heiss since Heiss should be accorded priority over Windemuth. See Application of Stempel. 241 F. 2d 755 (C.C.P.A. 1957); Application of Clarke, 356 F. 2d 987 (C.C.P.A. 1966). My problem with this argument is that Heiss had all documents presented here to show the Patent Office that Windemuth was not entitled to be cited as a prior reference. Although he did present this proof during the Price-Heiss interference to demonstrate reduction to practice and priority over Price, no such claim was made as to Windemuth. It is cited as prior art in the issuance of the Heiss patent with the evident acquiescence Heiss, and I see no need at this late date to depart from that determination by the Patent Office.

cross linkage is not required in such large quantities so that the resultant product is less expensive to produce.

The Windemuth Patent disclosed a process pursuant to which polyalkylene ether oxide compounds with at least two hydroxyl groups reacting with an excess of diisocyanates produced urethane products (foams, etc.) of the kind disclosed by Heiss. Windemuth refers to alkylene oxide and plaintiff contends, and all experts at trial concede, that alkylene oxide includes ethylene and propylene oxide. That Windemuth had in mind only ethylene and did not know or recognize the special qualities of propylene oxide is made clear by the fact, however, that throughout the patent disclosure reference is made to the finished product as "dissolving in cold water": "generally insoluble in water": "swell[ing] on contact with water"; "distinguished by a remarkable swelling behavior."18

Instead of an ethylene oxide condensate with two hydroxyl groups, Heiss' invention calls for a propylene oxide condensate with three or four hydroxyl groups.

¹³Plaintiff argues that since alkylene oxide includes both ethylene and propylene, the Heiss argument during the prosecution of his application that Windemuth disclosed only ethylene oxide misled the examiner, and, therefore, the patent ought to be invalidated. That argument has no merit. A patent may be invalidated or its validity undermined if the applicant misleads the Patent Office, see, c. g., Monsanto Company v. Rohm And Haas Co., 312 F. Supp. 778 (E.D. Pa. 1970), aff'd 456 F. 2d 592 (3rd Cir.), cert. denied, 407 U.S. 934 (1972). The yardstick, however, is whether the patentee has failed to give the Patent Office some vital information in connection with his application or has provided information in respect thereto which is erroneous or false. Nothing of that sort occurred in this in-Heiss argued that Windemuth disclosed only ethylene The examiner had the Windemuth Patent before him; he knew, as an expert, that technically alkylene oxide embraced propylene, as well as ethylene oxide, but read the patent as in reality disclosing only ethylene oxide. He was not misled or misinformed; the examiner merely agreed with Heiss' contentions.

This is essentially the only difference between the two processes, but that difference transferred hegemony in the production of polyurethanes from Germany to this The German developed urethanes won acceptcountry. ance in Europe but the foam products, because of their relative rigidity, sensitivity to water and to crystallization did not meet with much success in this country. Propylene oxide based foams are resistant to water sensitivity and crystallization and can be produced in both rigid and flexible form. Today, in the United States. one-half billion dollars in sales and one billion pounds of polyether derived propylene oxide foams, both rigid and flexible, are produced annually with flexible foam constituting over half the market. These are the foams covered in The Heiss Patent by the claims in litigation here.

The Sokol reference is not prior art. It is true his patent calls for a propylene oxide condensate, but it had nothing to do with the production of urethanes; the Hill Patent cannot be considered prior art; although he utilized a propylene oxide condensate, he obtained cross linkage through an entirely different process. Bayer led to Windemuth which has been discussed previously. as the polyether urethane constituted a great advance over polyester urethane, polypropylene based urethane constituted a still further advance in the art. Windemuth was prior art, but the Heiss process was novel and distinct and an advance beyond Windemuth's teachings. Therefore, the validity of The Heiss Patent cannot be successfully challenged as being unpatentable over Windemuth although its teachings do constitute prior art.

The Issue of Obviousness:

Since the only fundamental difference between Windemuth and Heiss was the use of propylene oxide, rather than ethylene, and since Windemuth makes reference to

alkylene oxide which includes propylene oxide, plaintiff argues that Windemuth rendered the Heiss invention "obvious". Thus, the question to be determined is whether the difference between The Heiss Patent and "the prior art[is] such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art. * * 35 U.S.C. §103. In resolving that question it is necessary to: (1) determine the scope and content of the prior art; (2) to ascertain the differences between the prior art and the claims at issue, and (3) to establish the level of ordinary skill in the technology in question. ondary considerations as commercial success, long felt but unsolved needs, failure of others, etc. might be utilized to give light to the circumstances surrounding the origin of the subject matter sought to be patented. indicia of obviousness or nonobviousness, these inquiries may have relevancy." Graham v. John Deere Co. of Kansas City, 383 U.S. 1, 17-18 (1966).

As has been indicated above, prior art had advanced from development of polyester based urethanes to polyother based urethanes by the time of the Heiss invention. Polypropylene oxide based urethanes being less subject to water sensitivity and crystallization than ethylene oxide based urethanes rendered the former an advance in the art. Yet, since propylene is a hemolog of ethylene, the question to be answered is whether it was not obvious on reading Windemuth to a person of ordinary skill in the art that the development of propylene oxide based urethane would follow as a next logical step. I find that the water resistant properties of polyether propylene oxide based urethanes which give them the great commercial superiority over polyether ethylene oxide based urethanes would not have been obvious to one of ordinary skill in the art at the time of the Heiss application.

Propylene glycols up to a molecular weight of 750 are soluble in water. Thereafter, water solubility decreases.

To substitute propylene oxide condensate for an ethylene oxide condensate and thereby conquer the problem of water sensitivity, therefore, was not clear. A person of ordinary skill in the art at the time of the Price, Heiss experiments would, it seems to me, reach the conclusion that there was no distinction in reference to water sensitivity and hydrolysis between using ethylene and propylene.

I am supported in the conclusion that the unexpected properties of propylene oxide were unobvious at the time to one of ordinary skill in the art, see Application of Courtright, 377 F. 2d 647 (C.C.P.A. 1967); Application of Petrzilka, 424 F. 2d 1102 (C.C.P.A. 1970), by Dr. Charles Price himself. In a speech, "How Chemists Create a New Product", he gave on February 2, 1961, published in the April, 1961 issue of "The Chemist", at pages 131, 133, Dr. Price said:

"While the claims [of the Windemuth patent] cover all poly (alkylene oxides), the disclosure and examples mention only poly (ethylene oxide). These German chemists [Windemuth, Schnell and Bayer] were working in a laboratory which led the world in developing polyester polyurethanes. In

¹⁴Jefferson argues that General cannot, in good faith, argue obviousness here as to Heiss, since it projected the Price Patent as valid in licensing the industry for the production of polyether propylene oxide based urethane foams under the Price Patent. Jefferson asserts General's contention undercuts the validity of the Price Patent and that it is taking that position only because its licenses are fully paid up. Thus, the argument goes General's assertions come within the unclean hands doctrine, and it should be barred from taking that position. It seems to me, however, that General has no choice other than to make this argument. Having admitted infringement, if the Heiss Patent is valid, General has only two basic defenses—prior conception and prior reduction to practice by Dr. Price and invalidity of the Heiss Patent—and is entitled to make both arguments despite their inherent inconsistency.

a patent filed over two years after our conception and reduction to practice, they had clearly failed to recognize the significant advantages of the propylene oxide unit over the ethylene oxide unit in decreasing water sensitivity and crystallization."

If the significance of propylene oxide in this respect was not recognized by Bayer, Windemuth and Schnell, who could hardly be classified as men of mere "ordinary skill", it is difficult for me to give credence to a contention that an ordinary journeyman would have understood that propylene oxide had these unexpected qualities in 1950 and 1951.¹⁵

I am compelled to conclude, therefore, that The Heiss Patent meets the test of obviousness under 35 U.S.C. §103.

The Issue of Prior Conception and Prior Reduction to Practice:

Since I have found that Windemuth does not invalidate Heiss' claim, the next question is to determine priority as between Heiss and Price. Mr. Heiss filed his application in the Patent Office on August 7, 1953, while Dr. Price's application was filed on September 23, 1953.

¹⁵ Moreover, Dr. Price and Mr. Heiss testified that in making their respective experiments in 1949 and 1952, they had to make in their own laboratories the propylene oxide which they used. This leads me to believe that propylene oxide did not become readily available commercially until the Price and Heiss disclosures. The testimony in the record is that it became commercially available in the 1950's, but Jefferson made no effort at trial to show specifically when in the 1950's this occurred. Therefore, while I cannot base any conclusion on what is only an unresolved suspicion, it is only fair to say that my conjecture that propylene oxide became commercially available on a wide scale coincident with Price-Heiss disclosures did produce added skepticism that its special properties would have been obvious to one of ordinary skill prior to the Price-Heiss disclosures.

The inventor who is first to conceive and reduce his invention to practice is ordinarily awarded priority. Boyce v. Anderson, 451 F. 2d 818 (9th Cir. 1971). Conception is the complete mental performance and all that remains necessary is construction. Priority over the application date of another is established when the experiment which predates the application is made sufficiently plain for those skilled in the art to understand it and reproduce The notebook in these respects constitute a contemporaneous record of the inventor's thoughts and actions. See Townsend v. Smith, 36 F. 2d 292, 295 (C.C.P.A. 1929); Summers v. Vogel, 332 F. 2d 810, 814 (C.C.P.A. 1964); Kardulas v. Florida Machine Products Co., 438 F. 2d 1118 (5th Cir. 1971); Langer v. Kaufman, 465 F. 2d 915 (C. C. P. A. 1972). However, unless a party can establish to a certainty as absolute as in a criminal case that his data of conception and reduction to practice preceded the date of the patent application, the application date controls. Karr v. Botkins Grain & Feed Co., 329 F. Supp. 411, 413 (S. D. Ohio 1970).

Dr. Price testified here, as in the interference, that he had the conception of making polyether propylene based urethanes in January 1949, and that he secured from General a grant to permit Dr. Herbst, a graduate student under him at Notre Dame, to work on the project and that in April, 1949, Dr. Herbst succeeded in producing the finished product. Produced at trial were reports of Dr. Herbst in memorandum form from him to Dr. Price which in turn were passed on to General. These records are disjointed and incomplete and do not make explicit that the process here in question was successfully concluded. Other than the unsatisfactory memoranda, initially prepared by Dr. Herbst for Dr. Price and transmitted by him to General, there is nothing to show reduction to practice prior to September 23, 1953 by Dr. Price. Dr. Herbst testified that more thorough

records were in a notebook he kept which apparently was destroyed by fire.

While this loss is unfortunate, the burden is on the plaintiff to prove that Dr. Price is entitled to a date earlier than that of his application. That burden has not been met.16 While it is unnecessary, therefore, for me to determine the date of conception and reduction to practice by Heiss prior to the August 7, 1953 date of his application to accord him priority over Price, there is a sufficient showing in the record on the basis of notebook recordings to establish that Heiss had the conception of polyether propylene oxide based urethane on September 14, 1951; and that he successfully performed an experiment producing a polyether propylene oxide based urethane foam in September, 1952. Thus, I conclude that Heiss' claims are entitled to priority over Price, insofar as General is now contending that the Price and Heiss processes are to the same invention.

General argues that the Heiss claims were triggered by knowledge of the issuance of the Price Patent; that the claims which are in issue here were first made a part of the Heiss application on February 6, 1959. Therefore, the non statutory doctrine of late claiming is urged as rendering the patent invalid and/or as entitling the claims to date only from the time when they were added to the application (February 6, 1959), rather than the date the application was filed. The rules of the Patent Office provide for "cancelling particular claims" and "presenting new claims" (Patent Office Rule 119). These claims, when allowed, take the original filing date of the application, even where there has been an intervening public use before the amendment has been made. See Coats

¹⁶In the Price-Heiss interference the attorney for Dr. Price indicated to him that he did not believe he would prevail on the issue of prior reduction to practice on the basis of proof of experiments at Notre Dame. I agree with this conclusion.

Loaders & Stackers, Inc., v. Henderson, 233 F. 2d 915, 924 (6th Cir. 1956); King-Seeley Thermos Co. v. Refrigerated Dispensers, Inc., 354 F. 2d 533, 539 (10th Cir. 1965). Moreover, the law seems settled, in the absence of some countervailing showing not made here, that the effective date of the claims of the continuous applications are those of the original application.17 General's argument in this regard is, therefore, rejected.

Plaintiff's argument of invalidity because of double patenting fails since I have found that the Heiss Patent is novel and distinct, and an advance in the art over In addition, its argument that Heiss is Windemuth. not the inventor is equally without merit. The transfer of the original Saunders-Heiss application to Heiss as sole inventor was made in conformity to the rules of the Patent Office and allowed by it. Plaintiff has presented nothing to undermine that allowance by the Patent

Office and its argument is rejected.

Finally, I find no illegality in the agreement between Mobay Chemical Corporation and Jefferson either under federal law, or, assuming New York law applies, under New York law. The agreement, as has been indicated earlier, gives to Jefferson proprietary ownership and title to The Heiss Patent. It is true that a condition of the agreement is either the issuance of one license under The Heiss Patent or institution of a lawsuit within a two-year time frame. This, however, does not condition Jefferson's right to the patent solely on the grounds that it sue.

The New York statute, N. Y. Judiciary Law §489 (Mc-Kinney 1968), provides that "... no corporation ... shall solicit, buy or take an assignment of, . . . a bond . . or other thing in action, or any claim or demand,

¹⁷A thorough discussion of this issue is Flocks and Neimark, Is There a Viable Doctrine of Non-Statutory Late Claiming as a Defense in Patent Litigation, 500 J. Pat. Off. Soc'y 676 et seq. (1968).

with the intent and for the purpose of bringing an action or proceeding thereon"

Plaintiffs rely primarily on American Optical Co. v. Curtiss, 173 U. S. P. Q. 654 (S. D. N. Y. 1971). Judge Ryan made clear the limited reach of his holding when he stated:

"It is true that the Agreement would not have been illegal under New York law if the University owned a patent and had made an absolute assignment of it to A. O., without conditioning the assignment upon the commencement of a suit by A. O. . . . Here, however, the assignment Agreement between the University and A. O. provided for and required A. O. to bring a lawsuit. Indeed, the Agreement appears to have been a contrivance solicited by A. O. to enable it to bring suit on behalf of the University, and the proposed suit was the very purpose and substance of the Agreement. A. O.'s intent to sue was not merely incidental and contingent. . . " Id. at 657 (Citations and footnote omitted.)

Moreover, in Fairchild Hiller Corporation v. McDonnell Douglas Corporation, 28 N. Y. 2d 325, 321 N. Y. S. 2d 857 (1971), the New York Court of Appeals said of \$489:

"We have consistently held that in order to fall within the statutory prohibition, the assignment must be made for the very purpose of bringing suit and this implies an exclusion of any other purpose. . . . The statute does not embrace a case where some other purpose induced the purchase, and the intent to sue was merely incidental and contingent." Quoting Sprung v. Jaffe, 3 N. Y. 2d 539, 544, 169 N. Y. S. 2d 456, 460.

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It follows from what has been said that the plaintiff's action is dismissed, and that defendant's counterclaim for an injunction enjoining plaintiff from further infringement of The Heiss Patent and for an accounting and damages is granted. Defendant's counterclaim for attorney fees and expenses is denied.

The foregoing constitutes the court's findings of fact and conclusions of law.

Settle Order.

Dated: New York, New York September 5, 1973

ROBERT L. CARTER U. S. D. J.

Judgment.

UNITED STATES DISTRICT COURT,

SOUTHERN DISTRICT OF NEW YORK.

[SAME TITLE.]

This cause was tried before this Court, Honorable Robert L. Carter, District Judge, presiding, Briefs and Findings of Fact and Conclusions of Law having been submitted by both parties, and the Court having written an Opinion, the same constituting its Findings of Fact and Conclusions of Law, entered on the docket September 6, 1973, it is hereby

ORDERED, ADJUDGED AND DECREED that:

- 1. Plaintiff's complaint is dismissed with prejudice.
- 2. Claims 3, 4, 5, 6, 8, 9, 10 and 11 of United States Letters Patent No. 3,102,875, issued to Herbert L. Heiss on September 3, 1963 for a "Polyurethane Reaction Product and Method for Making Same" and assigned to the Defendant Jefferson Chemical Company, Inc., are valid.
- 3. Claims 3, 4, 5, 6, 8, 9, 10 and 11 of United States Letters Patent No. 3,102,875 have been infringed by plaintiff.
- 4. Plaintiff is enjoined from further infringement of United States Letters Patent No. 3,102,875.
 - 5. Defendant is entitled to an accounting and damages.
- 6. Defendant's counterclaim for attorney's fees and expenses is denied.
 - 7. Costs are awarded to defendant.

New York, New York November 9, 1973

> s/ ROBERT L. CARTER United States District Judge

Judgment Entered Nov 9 1973

s/ RAYMOND F. BURGHARDT Clerk

Notice of Appeal.

UNITED STATES DISTRICT COURT,

SOUTHERN DISTRICT OF NEW YORK.

[SAME TITLE.]

Notice is hereby given that The General Tire and Rubber Company, plaintiff above named, hereby appeals to the United States Court of Appeals for the Second Circuit from the judgment entered in this action on the 9th day of November, 1973.

December 6, 1973

Watson Leavenworth Kelton & Taggart
Herbert Blecker
Attorneys for Plaintiff
100 Park Avenue
New York, New York 10017

To:

Kane Dalsimer Kane
420 Lexington Ave.
N. Y. N. Y. 10017
Attorney for Defendants

Excerpts From Trial Transcript. UNITED STATES DISTRICT COURT,

SOUTHERN DISTRICT OF NEW YORK.

GENERAL TIRE & RUBBER COMPANY,

Plaintiff,

vs.

JEFFERSON CHEMICAL COMPANY, Inc.,

Defendant.

68 Civ. 1227

Before:

Hon. Robert L. Carter, District Judge.

January 15, 1973 10:00 a.m.

Appearances:

Watson, Leavenworth, Kelton & Taggart, Esqs., Attorneys for Plaintiff, By John T. Kelton, Esq., Herbert Blecker, Esq., Robert E. Kosinski, Esq., James W. Badie, Esq., Frank C. Rote, Esq., of Counsel.

Kane, Dalsimer, Kane, Sullivan, Kurucz & Goldstein, Esqs., Attorneys for Defendant, By: John Caslin, Esq., of Counsel and Coffee & Sweeney, Esqs., By: James R. Sweeney, Esq., J. Robert Stapleton, Esq., Carl G. Ries, Esq., of Counsel.

(2) Mr. Kelton: Ready for the plaintiff.

Mr. Sweeney: Defendant ready.

The Court: Gentlemen, first I want to thank both sides for the excellent trial memoranda. I have almost reached

the point where I will be able to understand the chemical formula at the present time.

But I have set aside, as I indicated to you, six days for this trial. Some of the time will be cut into because I will have to take some pleas tomorrow and it may take an hour, and a sentence on Monday.

However, the trial will have to be concluded by the 22nd and, therefore, I will expect you to watch your time. I am prepared to work late at night or even on Saturday, but I expect you to watch your time and let me know whether or not the time allocation is sufficient.

Proceed.

Mr. Kelton: If the Court please, I am prepared and, indeed, I would suggest a brief opening statement, if the Court is prepared to receive it.

The Court: Of course. You may proceed any way you want.

Are you Mr. Kelton?

Mr. Kelton: Yes, sir, I am John Kelton.

The Court: You may proceed, Mr. Kelton, any way (3) you want.

Mr. Kelton: I will try to be as brief as I can and your Honor will understand that this is a summary, and not a summary of everything, indeed. I don't feel that it is required.

This is an action for declaratory judgment of invalidity and non-infringement. General Tire & Rubber Company is the plaintiff and Jefferson Chemical Company, Inc., is the defendant, and they have counterclaimed for infringement.

I might point out to your Honor that as we understand it, the only claims in issue of the Heiss patent in suit are Claims 3 through 6 and 8 through 11.

General concedes that these claims read upon what General does. So the primary issues before this Court

are validity and enforceability of Claims 3 through 6 and 8 through 11 of the Heiss patent in suit.

We contend that the patent is invalid and, of course, there can be no infringement of an invalid patent. That follows as a matter of law.

I would like to talk a little bit about the application for the Heiss patent, that is, the application filed in the United States Patent Office.

This application was in the Patent Office for ten (4) years, 1953 to 1963, but that doesn't mean that the Heiss right to the claims here in issue in this action received long or careful scrutiny; not at all. I will just briefly tell you why.

For the first five years, Heiss presented and argued and amended claims all directed toward a three-component system. This is the reaction between a polyol, one component, a polyisocyanate, second component, in the presence of a plasticizer, a third component.

That was for the first five years.

I would point out that none of the claims here in issue called for a plasticizer.

On December 30, 1958—that is some five years after the Heiss application was filed—there came an event which completely changed the direction of the prosecution of the Heiss application before the Patent Office, and that event was the issuance of Patent No. 2,866,744 to Dr. Charles Price.

A copy of that patent came to the attention of Mr. Upchurch who then on behalf of Heiss and the owner of the application was prosecuting the application before the Patent Office.

The significant thing was that was the trigger in this event of his seeing the Price patent, I think we must (5) conclude, was that there was no plasticizer in any claim. It didn't call for a plasticizer.

Upchurch moved quickly. This was the end of Decem-

ber 1958 that he saw the Price Patent. He moved very quickly.

On February 6, 1959, he copied the claims of the Price patent in order to say, "Well, my fellow invented that, too."

But I say to your Honor that this is the first recognition of a distinction on the part of Heiss or Price with regard to whether or not there was any difference or advantage of propylene oxide and ethylene oxide, one over the other. That came from Price and not from Heiss.

Then an interference was declared in July of 1959, testimony was taken and in March of 1962, the Board of Patent Interferences, who rules on interferences and not the examiner, who examines the application ex parte, held that Heiss, the Heiss application, could not support the Price patent claims.

In other words, they held that the limitation in the Price patent claims to a molecular weight of at least 600 was an essential limitation, essential feature, and it was one that was not shown by Heiss.

So the Board awarded priority to Price in March of 1962 on those patent claims.

(6) The Court: Awarded priority to Price?

Mr. Kelton: He awarded priority to Price of his Price patent claims for the reason that Heiss could not make those particular patent claims. We say "make," that is, he hadn't disclosed it.

Then not until August 13 of 1962 was what we call ex parte prosecution of the Heiss application resumed in the Patent Office. That is before the Patent Examiner. That is entirely apart from the Board of Patent Interferences.

In that resumption of ex parte prosecution, the Patent Office issued a paper which cited against the Heiss application, a patent which it issued to Windemuth against

the claims that were now in the type of those in issue here.

And in September of 1962, there was an amendment by the applicant Heiss through his attorney which contained representations as to the disclosure of this prior art Windemuth patent which we consider were inaccurate and we believe they were misleading.

Apparently, in acceptance of that representation, Windemuth was not further mentioned and after further communications between the examiner and Heiss' attorney, a notice of allowance was issued on December 11, 1962.

What I want to point out is that of the ten years (7) this application was in the Patent Office, only four months was given to consideration of the claims, such as are in issue here.

Far from a searching study and the exercise of expertise, I think this is a case which can most aptly be characterized in the same way Judge Learned Hand characterized a case he was hearing in 1924, and this is Lyon against Boh, and that is cited in the Footnote 3 in page 8 of our brief, and what he said is so short, I would like to read it.

"Courts have descanted upon the abuse again and again but the ant-like persistency of solicitors has overcome and I suppose will continue to overcome the patience of examiners and there is apparently always but one outcome."

On the matter of invalidity, I have referred before to this Windemuth patent. It is Windemuth Patent No. 2,948,691. It is prior art as to the Heiss patent under Section 102(e) of Title 35, which is the patent extension.

The fact is that Heiss now, although he has continually been aware of Windemuth since it was cited by the examiner on August of 1962 and has been aware

since quite early in this suit that General Tire has relied on Windemuth here and does rely on it, for the first time, not too long before we came to trial, Jefferson has alleged dates of (8) reduction to practice, that is completion of the invention, that he had an invention in hand prior to Windemuth's filing, U. S. filing date, that is, for the first time.

It is significant, I think, because there are three previous occasions when that could have been done and wasn't done.

First in the interference with Price, an interference is a conflict and priority, and part of the interference one has to file a preliminary statement, so-called, which is in the nature of a pleading and it precludes you as to proof of anything earlier than the dates you allege for conception or reduction to practice.

(9) The Court: Do you mind my asking you a question?

Mr. Kelton: No, your Honor, I would be pleased.

The Court: To be sure I understand your position, am I correct that you are alleging that Windemuth is prior art to the Heiss patent and that the claims which are made here are claims that are in the Price patent which were disallowed as being not claims in the Heiss patent at all? Is that the basis?

Mr. Kelton: So far as the Price patent is concerned— The Court: I want to be clear on the position of the Price patent.

Mr. Kelton: We do not and we cannot allege the Price patent as a patent itself, as art prior to Heiss. The Court: I understand that.

Mr. Kelton: But because Price was filed a month after Heiss, that is. I have often thought about the sameness of the words. I may get mixed up between Price and Heiss but I will try not to.

But what we are alleging and what we will seek to prove before your Honor is that in 1949 Dr. Price made

his invention and reduced it to practice and completed (10) it and he did not abandon it or suppress it in the meantime. So, therefore, that becomes a situation where by reason thereof it's our position that Heiss is not the first inventor.

And let me say that what Dr. Price did, he did the same thing that Heiss did except that he found that one had to or should have a minimum molecular weight of 500 or above for his linear piece which we will explain to you later. The linear portion of it that is.

Heiss' claims do not have nor did Heiss recognize any importance in minimum molecular weight. So Heiss' claims cover all molecular weights including those of Dr. Price so the latter includes something that came before it.

I would just like to say, in this interference with Price, in the Heiss preliminary statement which is an oath signed by the applicant, the earliest date alleged for reduction to practice of the invention was January 6, 1953, some eight months after Windemuth. ond instance is that on the citation of Windemuth by the examiner in August of 1962 Heiss did not assert that he was an earlier inventor than Windemuth and he could have. But instead he has avoided Windemuth by this incorrect and, I think, untrue representation as to what Windemuth disclosed (11) and then the third fact is that although in October 1970 Jefferson knew that General relied on Windemuth it wasn't until October of 1972 that they came through with the new answers which asserted dates which were presented as prior to Windemuth.

So this to me seems to give rise to two conclusions. No. 1, Heiss really has no date of invention prior to Windemuth and, secondly, that Windemuth was recognized as rendering Heiss invalid and we feel that Windemuth does invalidate Heiss and will ultimately be held to.

Now in this connection with regard to the misrepresentation, we will show here that Windemuth does disclose improvement of propylene oxide in the patent set forth in the Heiss claim.

It's our contention that Windemuth is in plain anticipation under Section 102(e) of the patent statute and for obviousness under 103.

We have another type of defense for invalidity and this is based on obviousness. It's based on what a skilled chemist, a chemist of normal skill in the art, would gain from reading a couple of items of the prior art and the patent law presumes that these are available (12) and usable against the inventor.

Now in this connection I will have to leave this to the experts to fill in and I may overstate myself but I will try to be conservative. There was an article in 1947 which shows reactivity of polyols which are one of the reactants important in going into the making of polyurethane resins. They show the reactivity of polyols with polyisocyanates to make urethane resins.

Bayer shows the employment of trihydroxy alcohols which Heiss' claims call for and is included within Heiss.

And this is to obtain what Bayer calls a reticulated structure. In other words, a cross-linked structure, sometimes called a network.

Bayer also teaches that ether linkages—and here we have polyether polyols—that ether linkages in the backhone do not interfere with a reaction with polyisocyanates.

Bayer teaches the practicability of the use of an excess of polyisocyanate.

Now the Hill patent also, that is No. 2,726,219, that has a date as prior art of August 24, 1951, the date under which the application for it was filed in the Patent Office.

(13) It disclosese reacting polypropylene glycol with a dihydroxy alcohol and with polyisocyanates. So really so far as Heiss is concerned all that Hill doesn't have is the use of a trihydroxy alcohol and it's our position that that is clear, the practicability of that when you want a reticulated structure is perfectly clear from the teaching of Bayer which was—the Bayer article I might say, in my view, was a milestone. And Hill clearly teaches the use of excess isocyanate which is a feature of the Heiss claims.

I would like to just touch briefly on one point. Jefferson in its brief argues the presumption of validity from the issuance of the patent. I would point out merely that this is a rebuttable presumption and that the circumstances here clearly rebut any weight being given to that presumption. I think the general thrust of the modern law is that the presumption of validity has substantially no weight. That instead it's a statement that it's the infringer's burden to show invalidity. It places the burden just as it's the plaintiff's burden or the patentee's burden to show infringement. (14) I think that is what it amounts to.

Now, as to the Price matter, I have already spoken to your Honor about it. As to Price, I would just like to say one thing:

In the Jefferson brief, it is suggested that the examiner considered the testimony in the interference.

I would say that in our view he did not and could not, particularly because of rules of practice of the Patent Office Nos. 106 and 107, and these set forth what may be cited as references and the only thing, in addition to the patents and printed publications, is personal knowledge of the examiner. In that case he must specify indeed what personal knowledge he had and the testimony of others as to events in the interference and certainly don't come within the knowledge of the examiner.

We contend that there is double patenting of Heiss over Windemuth. Mobay, we contend, was a common assignee of both and under the modern law, the later issued patent, the Heiss patent is involved. We treat this and cite cases on page 25 of our brief.

Now, as another defense we contend the contractual relationship between Mobay and Jefferson with regard to the enforcement of this Heiss patent was tainted and against public policy. The situation, briefly, is (15) that Mobay, as former owner of the Heiss patent, tried to license it and was unsuccessful; nobody would take it. Mobay felt for practical reasons, their business, that they didn't want to enforce it and so they transferred it to Jefferson, but it is the conditions under which it was transferred that also become very important.

In the contract it was provided that Mobay not only share its recovery to the extent of 40 per cent, but that Jefferson must, in order to prevent losing its entire purchase price and within two years, must license only polyol producers or sue foam manufacturers. That is how General came to be sued on the last day of the two years in Chicago. That suit, we contended, there was no proper venue of General there and, finally, before that motion was heard, Jefferson stipulated with us a dismissal.

Now, I think all these factors in this setup between Mobay and Jefferson make the agreement litigious, coercive and against public policy.

I might say Jefferson in their brief pointed to a litigation, which they had with Mobay, which I would submit to your Honor has nothing to do with this defense. They take the position that they have an airtight deal which can't be attacked. As I understand it, the Jefferson-Mobay litigation was, Mobay asserted that the (16) dismissal of the Chicago case was a breach of the contract and the whole thing was forfeited and that was fought

out in Delaware. That is an interpretation of the contract and nothing to do with public policy.

At any rate, your Honor, that is a summary of most of our defenses and there I will stop. If your Honor has any questions, I would be glad to answer them.

I think if there are technical questions they might better be answered with one with more expertise than

I have.

The Court: I will save them until then.

Thank you very much.

Mr. Sweeney: If it please the Court, I would like to withhold my remarks until after the plaintiff's case.

(19) HERMAN FRANCIS MARK, called as a witness by the plaintiff, being first duly sworn, testified as follows:

Direct Examination by Mr. Kelton:

Q. Dr. Mark, would you state your name and residence? A. My name is Herman Francis Mark. I live at 325 Ocean Avenue, Brooklyn, New York.

Q. Would you state your education and positions held?

Mr. Sweeney: Your Honor, defendant stipulates that Dr. Mark is qualified.

The Court: All right.

Mr. Kelton: I appreciate that, Mr. Sweeney, but I would say Dr. Mark is eminently qualified and I think it would be illustrative if we at least had his education and the positions held.

Mr. Sweeney: I did this only in view of his Honor has indicated that he wants us to stay within the time limit and I am trying to do it.

The Court: You may proceed.

Q. Go ahead, Dr. Mark. A. I obtained my Ph.D. in 1921 at the University in Vienna; moved to Berlin where I worked for six years as a research associate at a research institute.

(20) Then I went to industry for five years, to the I. G. Farben, where I worked mainly on polymeric materials.

I went back to Vienna as a professor at the University where I continued to study various types of polymers.

When Hitler invaded Austria in 1938, I immigrated to Canada where I was three years research manager of a paper company.

In 1940, I followed a call as a professor to the Polytechnic Institute of Brooklyn where I have been since then.

In 1965, I was emerited and I am now Dean Emeritus of the Polytechnic Institute of Brooklyn.

Q. Dr. Mark, have you prepared a brief resume of your education and positions held, including some of your editorships? A. Yes, I have.

Q. Is this it? A. This is it, yes.

Mr. Kelton: I offer in evidence as Plaintiff's Exhibit 3-A, a paper entitled, "Resume, Herman Francis Mark."

Mr. Sweeney: Is this the one that was supplied to us?

Mr. Blecker: Yes.

(21) Mr. Sweeney: I have no objection.

The Court: It will be received.

Mr. Sweeney: I would say this is not a complete list of all of his publications.

Mr. Kelton: It is not.

(Plaintiff's Exhibit 3-A received in evidence.)

By Mr. Kelton:

- Q. Now, Dr. Mark, do you have any publications? A. Yes.
- Q. About how many? A. In the last fifty years, I have published something like 500 papers. Of course, not alone, but with a rather large number of collaborators and about two-thirds of them had to do with polymeric materials of various kinds.
- Q. Have you prepared a selection of publications for purposes of this case? A. Yes, I have prepared a selection of those publications which have some contact with the subject matter of this case.
 - Q. Dr. Mark, I show you a paper. A. This is it.

Mr. Kelton: I offer in evidence a paper entitled, "Herman Francis Mark-Publications," as Plaintiff's Exhibit 3-B.

The Court: No objection?

(22) Mr. Sweeney: No objection, your Honor. I believe I have already seen it. That is the one that was given to me.

The Court: Mr. Kelton?

Mr. Sweeney: That was the one given to me earlier?

Mr. Blecker: Yes, it was.

Mr. Sweeney: No objection.

(Plaintiff's Exhibit 3-B received in evidence.)

By Mr. Kelton:

Q. Dr. Mark, have you received honorary degrees? A. Yes, again in the last fifty years during my work in various institutions with a large number of collaborators, I was privileged to receive a number of honorary degrees of universities in this country, in Europe.

Q. Can you tell me whether or not you have had any honorary memberships? A. Yes, I am an honorary mem-

ber of a number of scientific societies and academies, again in this country and in Europe and India.

Q. Can you tell me whether you have received any medals or awards? A. Yes, also.

Q. Dr. Mark, I show you a paper and ask you if that is (23) a list which was prepared by you. A. Yes, it is.

Q. It sets forth the honorary positions and memberships, academies, medals and awards? A. It does.

Mr. Kelton: 1 offer in evidence as Plaintiff's Exhibit 3-C, the document which has just been identified.

Mr. Sweeney: No objection.

The Court: It will be received.

(Plaintiff's Exhibit 3-C received in evidence.)

By Mr. Kelton:

Q. Dr. Mark, I understand you have been to China recently.

Can you tell me what was the occasion? A. Yes. I have received an invitation by the Chinese Academy of Sciences to visit a number of their research laboratories in the field of polymeric materials and I was there for three weeks in June and July.

Q. Have you received any other such invitations? A. Not from China. I am going to the Soviet Union in February on a similar invitation by the Academy of the Soviet Union of which I am a member also, in order to exchange opinions in the field of polymeric materials.

Q. Dr. Mark, are you familiar with the Heiss patent,

(24) Plaintiff's Exhibit 1? A. Yes.

Q. Can you tell me to what field of chemistry does that relate? A. The Heiss patent teaches the production of polyurethanes.

Q. That is to say, or do I understand you to say that it is polyurethane chemistry? A. Yes.

Q. Now, can you tell me what are the building blocks, if any, in polyurethane chemistry? A. Polyurethanes are produced when polyols and polyisocyanates are allowed to react with each other.

Q. The two components are, as I understand you, the

polyol and the polyisocyanate? A. Yes.

Q. Doctor, have you caused charts to be prepared to aid in illustrating, or if you think it is desirable, the chemistry which may be involved in this action? A. Yes. I have prepared a series of charts in the hope and with the intention to illustrate the various steps of the reaction between polyols and polyisocyanates and in the hope that it will accelerate the explanation of these reactions.

(25) Q. Might I ask you, then, if you would explain to the Court, polyols and polyisocyanates as a beginning? A. Yes. Could we have Chart A.

We are dealing in this discussion with organic compounds. Organic compounds are essentially built up by the elements of carbon, which is tetravalent, by the element of oxygen, which is divalent; the element of hydrogen, which is monovalent, and later on we will also have to add nitrogen, which is trivalent.

So, in order to repeat, the first line shows carbon-

Mr. Kelton: Might I interrupt, Doctor, just a moment.

The Witness: Please.

The Court: I think it would help if we marked for identification at this time as Plaintiff's Exhibit 4-A through 4-L—

Mr. Sweeney: Your Honor, may I reserve my objection.

Mr. Kelton: Yes, I am just marking them for identification.

The Court: He is marking them for identification at this point.

Mr. Sweeney: Yes, your Honor.

(26) Mr. Kelton: As Plaintiff's Exhibit 4-A through 4-L, the series of charts to which—or copies of the series of charts to which Dr. Mark has referred.

I might say that we have 4-A through 4-L, Λ being the first page and succeeding on through L. They are stapled together on a letter sized duplication.

I would ask that this be marked for identification as 4-A through 4-L.

(Plaintiff's Exhibit 4-A through 4-L marked for identification.)

Mr. Kelton: If your Honor please, we have a copy, which is stapled together, and each one is marked in pencil at the bottom of the page.

The Court: Thank you.

By Mr. Kelton:

Q. Sorry to interrupt you, Dr. Mark, but I thought we had better get that exhibit identified. A. That's perfectly all right.

Q. Go ahead. You can pick up wherever you want. A. The combination of hydrogen and oxygen leads to a group known as the hydroxyl group.

Its appearance in a larger molecule, when it is affixed somewhere to a larger molecule, then the suffix "ol" is added to the name of that molecule, and that is (27) the reason why we will meet and hear the words "polyol," "glycol," and others so often.

The importance of this group is that the hydrogen, which is here at the chart in red color (indicating), is a so-called active or reactive hydrogen which particularly reacts smoothly with the other component of the urethanes or polyurethanes, namely, with the isocyanate group, which we will discuss a little bit later.

On all those charts whenever a hydrogen is capable of reacting with an isocyanate group, it will appear in red color.

Now, the simplest compound between oxygen and hydrogen, of course, is water. As one can see, it has two active hydrogens and, in fact, it reacts very vigorously with isocyanates.

Of course, we are interested in the organic compounds which contain a hydroxyl group and the first of them is just the simple alcohol. It appears in the patent in column 1, line 61.

To every compound which appears in all those charts, there is a place where it appears in the patent. So this is column 1, line 61. In other words, it is one of the ols which are listed in the Heiss patent.

One can see that this has only one active hydrogen (28) and, therefore, it is called a monofunctional compound.

The next compound here is just another—the addition of another active hydrogen, another hydroxyl group, to the same backbone and we arrive now to a diol, which is called ethylene glycol. It appears in the patent in column 1, line 67, as another possible candidate for the reaction with isocyanates.

In this case, of course, we have two hydroxyl groups attached to a skeleton or backbone, which is known as ethylene. So, therefore, this material is called glycol or better ethylene glycol. It has two active hydrogens and it is difunctional.

The patent also discloses a little hit more complicated ols, triols, for instance, and on Chart C, in the upper part of Chart C, there is a very simple triol. It is nothing but the well-known glycerol and it has, of course, three active hydrogens and, therefore, it is a trifunctional compound.

In the patent, you will find it in Column 2 in line 19.

I may add here that the word "glycerine" is actually used in the patent instead of glycerol. That comes from a German version of the name of this compound so it is the same material.

(29) The patent even goes one step further and discloses in column 2, line 28, tetrafunctional ol, a tetrol, the name of which is pentaerythritol. It has four active hydrogens and is tetrafunctional.

So, we have here a first glimpse at the polyols. Every ol, which has more than two hydroxyl groups and as a consequence more than two active hydrogens, is called a polyol.

Poly is a Greek word and means nothing but many. It is just many.

So ethylene glycol is a polyol, glycerine is a polyol and this pentaerythritol is a polyol. It is a generic term for these compounds.

These are very simple ones and a little bit later, we will have to come back to this same disclosure here because the patent familiarizes us with much more complicated and, in fact, much more important polyols, but I would like for a moment now to go to the other integral component, namely, to the isocyanate—and if I could have Chart J?

There one finds in the first line, the title of this chart, "Formation of a Urethane Linkage." Now we are where we want to be at the end, namely, at polyurethanes.

In brackets, there is a capital U because in the lower part of the chart for brevity and simplicity, we will (30) replace the urethane linkage just by the letter capital U.

What happens if an isocyanate group which is depicted here in the second line of Chart J, if this group reacts with a hydroxyl group, then what happens is that the hydrogen of the hydroxyl group migrates over here to the oxygen, in other words, this oxygen has a higher

affinity for the hydrogen than this oxygen, and it gets this combination in which a new bond, this bond here (indicating), has been established between the two molecules which have been separted before, so a condensate is formed. Two individual separate molecules have now combined with each other.

The patent mentions very many isocyanates. The patent mentions very many ols and, therefore, I have here left or in the form of four little dots indicated that there is another skeleton, there is a carrier, there is an inert part of the molecule which carries this reactive group.

There we get a urethane linkage.

Now, this happens, for instance, if we have here ethyl alcohol. In the patent, you will find the reference to the reaction of an isocyanate and a hydroxyl group in column 1, line 51 and in column 2, line 44.

The urethane linkage, you will find, or the word "urethane, you will find in the title of the patent and (31) also in column 1, line 28.

Now, that happens if a monoisocyanate and a monohydroxyl carrying molecule are allowed to react with each other. Each of them has only one arm outstretched and if they grab each other, one gets a pair and that is the end of it; but if one instead uses a diol of the type which has been already discussed, such as diethylene glycol, and a diisocyanate, in other words, a molecule which has two isocyanate groups and a molecule which has two hydroxyl groups in this formula here, the hydrogen, which is attached to the nitrogen after the reaction. was attached to this oxygen before the reaction and when the glycol and the diisocyanate reacted with each other, a bond was formed here and "here" means between the CO group and the oxygen atom and the resulting adduct has now again two functional groups, on one side an isocvanate group and on the other side a hydroxyl group. They are left over.

When we go to the abbreviation here where we abbreviate this little bit clumsy group with U, we arrive at an adduct of the diisocyanate.

What we do now is we operate with materials which have two outstretched arms and can grab each other. In this case, a chain forms. The reaction is not over after one interaction, but there is always reactivity left and the (32) reaction can go on.

What eventually results, if there are enough molecules of this type in a vessel, and, of course, there are always very many molecules available, a polyurethane is produced; a polyurethane which consists of the backbone of the disocyanate, the urethane bond, the backbone of the glycol and other urethane bonds all along the chain.

Then we are now here (indicating) at the word "polyurethane" which appears in the title of the patent.

The word "diisocyanate" which is the word which teaches us in the patent to use a diffunctional isocyanate can be found in column 2, line 51.

We will remember that the patentee, Mr. Heiss, also discloses triols, the glycerol which we had in column 2, line 19, and which we have in Chart C on the top.

If such a material which has three active hydrogens is reacted with a diisocyanate out of every active hydrogen, a chain grows and Chart K shows what happens in this case.

This chart illustrates the formation of branched polyurethane chains from trifunctional polyols.

The trifunctional polyol in this specific case is glycerol and we react it with a certain diisocyanate of which we have not in detail disclosed the backbone because it doesn't make any difference in terms as to whether or not (33) the reaction occurs.

Then, of course, out of each of these reactive hydrogens, a chain grows and since there are more tri-

hydroxy compounds, more triols in the mixture, eventually one arrives at a polyurethane network.

As long as there are two outstretched arms, one always gets chains, if I would have another arm to connect with another chain, then one eventually would get a network.

That is the importance of the use of a triol or a tetrol which will become evident as we go through the rest of the patent.

Summarizing, we can say that we have now attempted to illustrate in the case of simple polyols of a glycol, glycerol, pentaerthritol, their reaction with disocyanates.

But the disclosure teaches many other polyols. There exists a very large number of polyols, all of which react with isocyanates or with polyisocyanates, and all of which are commercially available and of considerable importance.

Maybe I should start the discussion and illustration of these more complicated polyols by referring to column 1, line 66.

In this line, it is said that alkylene oxide condensates of hydroxyl compounds, of these hydroxyl compounds, will form a number of polyols. In Chart B, I have attempted (34) to illustrate this.

The first question is, what is an alkylene oxide?

Well, it will be remembered that this group -C-C-HH in the first line of Chart B, is the ethylene group. We have this ethylene group already in Chart A at the bottom.

Therefore, we have here the same ethylene and glycol which we have at Chart A at the bottom and which the patent mentions in column 1, line, 67, and which has, of course, two reactive hydrogens, two hydroxyl groups.

Now, in some way it is possible to remove water from this difunctional molecule and to arrive at a new compound.

The compound, which has a ring, it has a ring of three atoms, two carbon atoms and one oxygen atom, and you can see that the backbone is ethylene and since oxygen is here combined with it, this material is called ethylene oxide. Ethylene oxide is an alkylene oxide because ethylene is an alkylene residue.

Now we go to the next alcohol in the series of alcohols, the propyl alcohol. The propyl alcohol is mentioned in the patent, column 1, line 66, right next to the ethyl alcohol.

Now we can see that the propyl alcohol and the ethyl alcohol in the next-to-the-last line of Chart A are (35) very closely connected.

If I add to the ethyl alcohol one CH₂ group, then one gets the propyl alcohol and if one adds another one, one gets a still heavier alcohol and, in fact, in the patent in column 1, line 60, several of these alcohols are enumerated; compounds of this kind which are very closely related to each other and only distinguished by one CH₂ group are called homologs.

This word will be used later, so that propyl alcohol is a homolog of ethyl alcohol and fittingly in the disclosure, it is actually mentioned directly next to ethyl alcohol.

We can do to propyl alcohol the same thing what Chart A, the last line, illustrates for ethyl alcohol, namely, it may have two hydroxyl groups and then, of course, it is a diol and it is a diffunctional material and this backbone is propylene instead of ethylene so that this material is called propylene glycol and, of course, it is also mentioned in the patent right next to the ethylene glycol in column 1, line 67.

So it is clear that ethylene glycol and propylene glycol are homologs.

One can do to propylene glycol the same thing what we have illustrated up here with ethylene glycol, namely,

(36) one can close this ring by removing water and then, of course, one arrives at another alkylene oxide where now the backbone is not ethylene but propylene and this is, therefore, called propylene oxide.

So ethylene oxide and propylene oxide are homologs. I may say briefly that actually the commercial production of the ethylene oxide and propylene oxide does not go by removing water from the glycol but by some other way.

This is just not to create the impression that this is actually the commercial way in which these two alkylene oxides are made.

So that we have, so to speak, a new ingredient now, the alkylene oxides. These alkylene oxides, of course, can now be reacted with what we know already, namely, with the glycols and we go back to Chart C, the lower part of Chart C.

There is illustrated the reaction of ethylene glycol on the left side. Here we have the ethylene glycol and this is now reacted with ethylene oxide. One ethylene glycol and one ethylene oxide, that is.

Again, what happens is that the hydrogen moves over here and this adduct is formed.

(37) Of course, it still has two reactive hydrogens because one is left on this side and the other one is newly created over here and now the two molecules which originally had been separated from each other are combined by an oxygen atom, the oxygen atom being difunctional, the two outstretched arms, and can grab one of these residues with one arm and the other with the other arm and one gets a chain.

The bonds of this type are called ether bonds and we will have much to do with polyethers.

So, polyethers are, for instance, obtained by the reaction of polyols in this specific case of a glycol with an alkylene oxide.

You will find this reaction, actually the result of this reaction, in column 2, line 1, where we have the word "diethylene glycol."

We have two ethylene residues and we have two hy-

droxyl groups so this is the diethylene glycol.

The patent tells us, one can also go further, one can make the triethylene glycol in the same line. I think we can go further than that and in Chart D is illustrated what the patent teaches us.

In column 2, line 2, it is noted. There, the patent uses the word "polyethylene glycols" and Chart D (38)

illustrates how they are made.

One starts here with an ethylene glycol and then allows it to react not with one ethylene oxide but with more than one ethylene oxide and then first one ethylene oxide adds, another hydroxide group forms and it continues and we get a linear chain of polyethylene oxide or polyethylene glycol.

We can call that a polyethylene oxide because of the ether bonds and we can also call it a polyethylene glycol

because of the end groups.

You will find in the literature both names used. Since we are interested, and also since the patent is particularly interested in the reaction of the end groups, we will usually find the word "polyethylene glycol," In fact, in column 2, line 2, this expression is used.

Then, evidently, what one can do with ethylene oxide, one can do with propylene oxide which the patent discloses. That is in terms of column 2, line 4, where it

talks about polypropylene glycol.

If we can have a look at Chart E. Chart E illustrates the preparation not of a polyethylene glycol but of a polypropylene glycol and it starts in the first line with a propylene glycol, with a monopropylene glycol, a small molecule which appears in the patent in column 1, line 61, (39) reacts it with propylene oxide and then one obtains the dipropylene glycol.

Again, the hydrogen migrates over here and we get an ether bond so the dipropylene glycol is an ether polyol. The dipropylene glycol is mentioned in the patent in column 2, line 3.

Just as before with the ethylene oxide, one can do the same thing here with propylene oxide, namely, using more than one molecule, and one arrives in this manner to a polypropylene oxide or polypropylene glycol, which the patent mentions in column 2, line 4.

Those are all linear condensates of alkylene oxides with glycols. They must be linear because a glycol has only two hydroxyl groups and, therefore, all we can get are chains.

In one case we have polyethylene chains and in the other case, we have polypropylene chains.

But, of course, the patent has told us before that one cannot only use diffunctional alcohols but also trifunctional, that is the glycerol which appears in column 2, line 2, on—or rather line 19, I'm sorry.

And the next chart, namely, the Chart G, shows what happens if one allows glycerol to react with ethylene oxide because glycerol has three hydroxyl groups in three directions, addition of ethylene oxide takes place, to the (40) left, to the right, by these two hydroxyl groups, and on top or vertically by the third hydroxyl group.

The bonds between the individual ethylene oxides are all ether bonds, indicated here by these red arrows, and at the extremity of each chain, there is a reactive hydrogen and I just see that an error was made here when this chart was drawn, because, evidently, this hydrogen on top here being connected with oxygen is a reactive hydrogen. This is a hydroxyl group.

So maybe the record should show that a correction was made here and that this black H was converted into a red H. We get a trifunctional polyethylene oxide or polyethylene glycol.

Again, this is mentioned in line 20 and 21 of column 2, where it is said that alkylene oxide condensate of glycerine—this is an alkylene oxide condensate of glycerine, it is the material which is mentioned here in column 2, line 20 and 21.

For the brevity of eventual later discussions, the formula has been just rewritten and instead of putting in all of this somewhat bulky CH₂ groups, I have here put in ET for ethylene and GL for glycerol. But it is the same formula only in a somewhat abbreviated form.

The same thing, of course, can be done with (41) propylene oxide which is the other alkylene oxide which is discussed here and Chart H shows what happens.

We have here the glycerol trifunctional, so we get three chains growing out with ether bonds and with three functional hydroxyl groups, a branched polypropylene glycol, which is also a polyether polyol and in this case, a trifunctional polyether polyol.

This is a very important class of polyols which the Heiss patent discloses here in terms of possible candidates for the reaction with isocyanates.

They are generic terms, all polyether polyols, polyether because the bonds between the individual building stones are ether bonds, and polyols because they have two or more terminal hydroxyl groups.

Unfortunately, they are not the only polyols which can be used in order to build up polyurethanes.

There is another big class of them where an entirely different reaction is used in order to build up the long chains but where the functionality has exactly the same consequences as it had before.

These materials, of course, are also disclosed in the patent and they come about by the reaction of something which we haven't had until now. by the reaction of an acid and alcohol.

(42) The acid is mentioned in the patent, column 2, line 9, and now we go to the last chart, to the Chart I, and this chart here illustrates the reaction of alcohol. We know already ethyl alcohol without acid, but we haven't had an acid yet in these charts.

An acid is a compound which does not only contain a hydroxyl group but also a CO group, so characteristic-

ally for an acid is a CO group here.

If one removes water from a monofunctional alcohol and a monofunctional acid, this acid happens to be just acetic acid, a standard acid used in making vinegar. Then one gets an adduct.

Here is the alcohol component and here is the acid

component and the water is eliminated.

This group here, the OCO group is called the ester group. In other words, we can build up the polyols either with the aid of ether bonds or with the aid of ester bonds, two big groups of polyols exist polyether polyols and polyester polyols.

The backbone is different. The reactivity of the end

groups is the same.

All the principles of linearity and cross-linking which hold for the polyether polyols also hold for the polyester polyols.

(42a) The next step, of course, is now to use a difunctional alcohol and a difunctional acid and then we get a long chain where we have alternated the alcohol and the acid where ester bonds are involved but where, again, of course, two terminal hydroxyl groups are left.

(43) Now in the patent the ester polyols are also, of course, disclosed and specifically in Column 2, line 8 and in Column 2, line 8 to 10. Then it says in Column 2, line 8 to 10, an ester prepared from one mole of a dibasic acid. that is this acid here, and two moles of a glycol forms a polyester.

The word polyester appears in Column 2, line 9 and

10.

Those are the substantially or all the important polyols in terms of their structure and in terms of their preparation. Of course, what one can also do with polyester polyols which I haven't actually written down in order not to make this discussion too long-winded, if you use a trifunctional alcohol one does not get a linear polyester but one gets a branched polyester and eventually a cross-linked polyester, just exactly in the same manner in which one gets it with the aid of alkylene oxides.

If one attempts briefly to summarize the teaching of the patents starting with Column 1, line 57 and ending in Column 2, line 33, this teaching is devoted entirely to the enumeration of a large number of polyols. All of them have reactive hydrogens either at two or three, or four ends but they do have different backbones.

The patent teaches us that they are all suitable (44) because in line 58 it says "All as illustrative examples of suitable hydroxy compounds". So they are all suitable. They all react with isocyanate groups, polyester or polyether and those with propylene oxides and ethylene oxides.

The patent now continues in Column 2, line 33 to discuss a group of substances which are called thioure-thanes. I don't think it is worthwhile to discuss them in detail. They are actually never practically used.

However, then follows an important teaching starting in Column 2, line 44 and ending in Column 3, line 9. This is information on the isocyanates which can be or may be used. That is what the first sentence says:

"Illustrative examples of suitable isocyanates which may be employed" and then again there is a large number of different isocyanates, mono, di, triisocyanates enumerated. Fortunately, only two of them are truly important for the purpose of our discussions and they are shown in Chart L.

First of all in a diisocyanate we want to see two isocyanate groups and here they are and then there is a backbone, a core or a skeleton which holds those two isocyanate groups which is of a somewhat complicated (45) character. It really doesn't matter very much what it is. Chemically, if needed later, I think all witnesses will be easily capable of disclosing details but it really doesn't matter. It is a commercially important diisocyanate and it is disclosed in the patent, Column 3, line 1 under the long-winded chemical name which is always abbreviated as MDI.

So this is MDI, disocyanate. The other important disocyanate is shown underneath on Chart L and is mentioned in the patent in Column 2, line 68 and 69.

We want to have two isocyanate groups which we have and one can see this is now another skeleton or core but the reactivity of these two groups is substantially the same as the reactivity of those two. So for the building up of polyurethanes this material abbreviated called TDI is a useful as MDI.

As T said it already, it is mentioned in Column 2, line 68 and 69.

I think that concludes the enumeration of the two integral components of a polyurethane, namely of the polyols and of the polyisocyanates as far as they are disclosed in the Heiss patent.

Q. Dr. Mark, in your discussion you used many times the term functionality in terms of being monofunctional, (46) difunctional, trifunctional or tetrafunctional.

Could you just simply explain what you mean by functionality? A. Well, functionality refers to the active or functional groups. We are dealing here with two types, with the hydroxyl groups and with the isocyanate group. If a molecule carries two of those groups it is diffunctional. If a molecule carries three it is trifunctional and if a molecule carries four, it's tetrafunctional and so on. So it's

really the number of reactive or functional groups which one molecule still has at its disposal for further reactivity.

Q. Doctor, in discussing Chart H, you indicated that

GL stood for glycerol, I think. A. Yes.

- Q. Do you mean glycerol as it is with three terminal hydroxyl groups or do you mean as the glycerol backbone? A. It stands for the glycerol backbone, for the glycerol residue.
- Q. In the same chart you have used PR to mean propylene? A. Yes.
- Q. What do you mean that to refer to? A. This is the propylene residue.
- (47) Q. In other words, the propylene backbone, would that be correct to say? A. Yes, sir.
- Q. And in some cases you have used ET for ethylene? A. And this, of course, also is the ethylene backbone C_2H_4 .
- Q. With regard to the Heiss patent as to which you have explained the chemical terms, let me ask you a couple of questions.
- Dr. Mark, what does the patent teach as to the suitability of polyesters, polyethers as the polyol component? A. The patent teaches as I have already mentioned, that all the materials which are mentioned as illustrative examples of suitable hydroxy compounds from Column 1, line 57 to Column 2, line 33 all are suitable.
- Q. I am sorry, I guess I didn't quite realize that when you testified— A. I may have mentioned it a little bit too fast.
- Q. Let me ask you, along this line, what does the patent teach as to suitability of diols, triols or tetrols as a polyol?
 - Mr. Sweeney: I think that in expert situations leading questions are helpful but I ask that they be kept (48) to a minimum. That is pretty leading.

The Court: All right.

Mr. Kelton: I fail to see wherein that is.

The Court: I don't think that Dr. Mark had understood that you had asked him a question.

Q. Did you understand the question, Doctor? A. Yes, I understood the question. I was just aware of the fact there was an objection.

Q. Does the patent teach anything as to the suitability of diols, triols and tetrols as the polyol component? A. The patent teaches that they are all suitable.

Q. Does the patent have any teaching with regard to the ratios of isocvanate and polyols?

The Court: I am having difficulty hearing now, Mr. Kelton, and I am sure Mr. Sweeney is.
Mr. Kelton: I will repeat it, your Honor.

Q. Does the patent have any teaching with regard to the ratios of isocyanates, on the one hand, and polyols, on the other? A. Oh, yes. I may have created a misunderstanding as if the teaching of the patent would be only restricted to the polyols and to the polyisocyanates; the teaching goes on.

(49) The next question is in what ratios should one use these components, whatever they are. This is specifically disclosed in Column 3, line 28 to 55. If one reacts, for instance, difunctional components with each other, as for instance here in this little string of beads, where we could imagine the white beads are a glycol and the blue beads are a diisocynate, then it is evident, if I have an excess of a diisocyanate of 5 to 4, the end groups will be isocyanate groups.

If I have an excess of glycols 5 to 4, then the end groups will be the white balls and will be glycols or would be hydroxyl groups, excuse me, I was wrong, would be hydroxyl groups, so the ratio with which the two components are reacted with each other are of importance

because they determine what kind of groups we do have at the ends of a linear chain or at the ends of the chains which form a network.

Realizing, of course, this importance, the teaching of the patent in the paragraph which starts 3-28 and goes to 3-55, teaches that an excess of the isocyanate groups should be used in reacting—whenever one reacts a polyol with an isocyanate, one should see to it that the number of isocyanate groups in the reacting system is larger than the number of hydroxyl groups.

(50) There are specific ranges given in the patent, but I think the essential thing is that the patent teaches an excess of isocyanate groups over hydroxyl groups.

There is another component, which the patent teaches as being used and as belonging to the invention, and that is in Column 4, line 1, where it says again:

"As illustrative examples of non-reactive organic liquids and solids which may be used in the practice of this invention . . ."

and then he enumerates a large number of substances of this type. These materials are also referred in the teaching of the patent as plasticizer and the word plasticizer appears, for instance, in Column 3, line 70 and then again in Column 4, line 25.

These plasticizers are characterized as nonreactive organic liquids and solids and specifically it is demanded that their boiling points should be above 200 degrees Centigrade and preferably above 250 degrees Centigrade.

So that we have at the end of the teaching, before the examples are introduced, a disclosure of three components of which two are reacted in the presence of the third. The two are the polyols and the polyisocyanates and the third are the non-reactive organic liquids or, as (51) it is also called, plasticizer.

Now, then, one more point should be mentioned in terms of the teaching of the patent up to the point where the examples are being enumerated and that is how is the reaction brought about. If one mixes materials of this type, some of them are liquids, some of them are solids, at room temperature nothing happens or a little happens or in some cases a little happens and in other cases nothing happens.

Therefore, in Column 3, line 49, it is disclosed that

the reaction is achieved by heating.

In the next line it is disclosed that it also can be effected by means of catalysts. Now, catalysts are by definition materials which are added in small quantities to a reacting system and accelerate the rate of the reaction which is supposed to take place without actually entering the reaction themselves. So that if the reaction between a polyol and a polyisocyanate and a plasticizer is carried out in the presence of a catalyst, then we have actually another component. So, these are substantially the teachings of the patent up to the beginning of the examples.

Q. To go back to this diol, triol, tetrol matter for just

a minute, Dr. Mark— (52) A. Yes, sure.

Q. Can you tell me whether or not the patent indicates any preference of a diol over a triol or vice versa? A. No, it doesn't.

Q. Now we can proceed with the patent for a moment. Does the patent contain illustrative examples? A. Yes.

The Court: The question was:

"Does the patent contain illustrative examples?", Mr. Sweeney.

Mr. Kelton: I am sorry, your Honor.

A. Yes. Actually the patent starts with the disclosure of the examples in Column 4, line 29 up to Column 9, lines 74. I would submit that for the simplicity of the presentation one might group the examples, first, in two and a little bit later in three groups.

Q. Dr. Mark, I note the part they are grouped under is "Manufacture of Substantially Bubble Free Gels and Resins" as to Example I, and then under Example XXII in Column 9, the heading: "Manufacture of Resinous Foams". A. That is correct. That is a grouping which the patentee himself suggests. Twenty-one examples are devoted (53) to the preparation of substantially bubble-free gels and resins and four to the preparation of foams.

Q. What does bubble-free mean, as you understand it? A. Yes, if one takes the three ingredients of any of the first 20 examples or 21 examples and mixes them together in a flask, increases the temperature in order to get the reaction going, something may be split off during the reaction. There are impurities in the ingredients which cause side reactions and the impurities and also at the high temperature even split off fragments create bubbles. They are gases and as the viscosity increases very rapidly during the reaction, the bubbles get trapped. the end, when there is a piece of sticky resinous material, rubbery sometimes, sometimes brittle which contains hubbles; and this for many applications is very undesirable. It is undesirable for coating and it is particularly undesirable for an electric insulation. It is undesirable for an adhesive and, therefore, the attention of the patentee is directed—by himself, of course,—to substantially bubble-free gels and resins.

Q. Dr. Mark, could you comment, first of all, on a group which I will select of the examples, namely, Examples I through VIII.

(54) Would you discuss those examples? A. Yes, as a group.

Examples I through VIII I think can be summarized as a group because they use all the same triol, the same polyol.

It is a polyol called triethanolamine as can be seen in Column 4, line 37 and it is a compound which we have not yet encountered.

- (55) Q. Dr. Mark, in Example 3, there is diethanolamine, is there not? A. Yes.
- Q. That would be a diol rather than a triol? A. Diethanolamine, the other use triethanolamine.
- Q. You go ahead. A. I don't think it is necessary to go to the chemistry; whenever it is needed, we can do that. One is a triol and the other is a diol.

The diethanolamine acts like glycol and the triethanolamine acts like glycerol.

Then, as far as the other component is concerned, namely, the diisocyanate, Examples 1, 2, 3, 4, 5 and 6 and 8 all use TDI, that is the material which appears first on column 4, line 38, and it is called m-tolylene diisocyanate. It is the material which we have on Chart L abbreviated as TDI.

In Example 7, the patentee uses the other diisocyanate, namely, MDI, just in order to establish whether it can be used.

All examples use a non-reactive organic liquid as plasticizer.

Example 1, 2 and 3 use a specific material disclosed earlier in the patent, which appears in Exhibit 1 (56) in line 39 and is referred to as chlorinated hiphenyl.

It is a well-known commercial compound and contains chlorine and is an aromatic material used for many purposes. It is a high boiling material.

In Example 4, another plasticizer is used. It appears in column 5, line 1 and 2 under the name chlorinated isopropylbiphenyl, which is just another high boiling inert substance.

Example 5, actually 7 runs were made in Example 5, each one with a different non-reactive organic liquid.

Here in this example in column 5, line 12, these non-reactive plasticizers are referred to as solvents, which just means that they do not interfere with the reactions they are not supposed to interfere with the reaction and,

in fact, do not. There are all kinds of different materials.

Example 6 goes back to chlorinated biphenyl.

Example 7, which works with a different disocyanate, namely, MDI, also uses hiphenyl.

Example 8 also.

In other words, summarizing, one might say that two different polyols are explored in those examples, namely, the diethanolamine and the triethanolamine.

Two different disocyanates are studied and some three different—more than that—oh, quite a few different (57) plasticizers are studied because the plasticizers in Example 5, although they are all chlorinated biphenyls, contain different amounts of chlorine, so each of these plasticizers is a different chemical compound.

So there is a very elaborate study of this plasticizing

component carried out in those eight examples.

Q. In these eight examples, are there substantial variations in things other than the thing that I will call a plasticizer or high boiling substance? A. There are differences in the ratio between the two reactive ingredients. There are differences in time of the reaction.

There is not very much difference in the temperature which is used, but, in any event, these examples show that there was a considerable amount of work done in order to find out what happens with different plasticizers, with those two polyols and with those two diisocyanates.

Q. Now, Dr. Mark, with regard to Example 5, I think you said something to the effect that the high hoiling liquids were indicated as solvents. A. Yes.

Q. And do not enter into the reaction. A. That's correct.

(58) Q. What kind of a reaction were you talking about? A. The reaction between the polyol and the disocyanate.

Q. You mean the chemical reaction? A. The chemical reaction.

Q. Would you comment on Example 9, Doctor? A. Example 9 is a different kind of test in that castor oil is used. Castor oil, again without going into the details, is a triol.

In other words, castor oil has on its backbone three reactive hydroxyl groups. They come from glycerol. One part of the castor oil is glycerol and the other part of castor oil is a long chain acid, which carries these hydroxyl groups, so that after the ester between glycerol and the acid, one has these three active hydroxyl groups, but this is a polyester, this is not a polyether, so as, for example, Examples 1 to 8 operate with a polyether polyol, Example 9 operates with a polyester polyol.

It uses TDI, as the others. It uses chlorinated biphenyl,

as the others.

The result was a flexible bubble-free resin similar to the results of some of the other examples.

Q. You say castor oil is a polyester polyol? (59) Q. Would it be correct or incorrect to refer to it also as a polyester triol? A. Yes. In fact, it is specifically a polyester triol.

Q. With regard to Examples 1 through 9, can you tell me whether or not any of these examples involve a condensate of propylene oxide and a polyhydric alcohol having from three to four hydroxyl groups? A. None of them does.

Q. Do any of Examples 1 through 9 involve a condensate of alkylene oxide and a polyhydric alcohol having

three to four hydroxyl groups? A. No.

Q. Now, can you tell me whether any of the examples in what I understand to be the bubble-free group, namely, examples 1 through 21, involve a condensate of alkylene oxide and a polyhydric alcohol having three to four hydroxyl groups? A. Yes, of course, all examples from 10 to 21.

Q. 10 through 21? A. Yes.

Q. What are the illustrations of those examples, 10 through 21? Would you start with Example 10, please? A. Example 10 studies the adduct or condensate of a (60) certain amount of polypropylene oxide with 1 mol of glycerol in its reaction with TDI.

First propylene oxide and glycerol are reacted with each other and then TDI is added and reacted in the presence of chlorinated biphenyl, about 50 percent by weight. One obtains a tough, rubbery and resinous gel.

This is a reaction of an alkylene oxide, glycerol adduct which is a triol. Specifically, it is a polypropylene compound.

There is an excess of NCO over hydroxyl of about 2 to 1. That is Example 10.

Should I go on to Example 11?

Q. Yes, if you would, Doctor, please. A. Sure. Example 11 reads as follows:

Meta-tolylene diisocyanate, which is TDI, was reacted with the following alkylene oxide condensates of glycerine. So a number of alkylene oxide condensates of glycerine or glycerol were prepared first.

Two of them were prepared with propylene oxide and two of them were prepared with ethylene oxide.

The difference in each pair, that is, the two propylene oxide condensates differed in molecular weight from each other and the two ethylene oxide condensates differed in molecular weight from each other.

(61) Each of these preparations was reacted with TDI in the presence of about 50 percent by weight of plasticizers.

So this example evidently wants to establish two things:

First, if I take either ethylene or propylene oxide for itself, is there a difference if I use a different molecular weight.

Second, if I use comparative molecular weights, is there a difference between the use of ethylene oxide and propylene oxide?

So is the propylene oxide condensate of a certain molecular weight different from the ethylene condensate of about the same molecular weight.

It is a double-study really. It studies the molecular weight on one component and also studies the relative merits of two components.

(62) Q. Doctor, when you refer to molecular weights, does that have any relation to the expressions in this example 11, for example, 3.1 mols of propylene oxide with one mol of glycerine? A. Yes, and if glycerine has, as it has, three reactive hydroxyl groups, out of each a polypropylene oxide chain grows.

If I have three mols or whatever it is, 3.1 mols as compared with 13.2 mols, the molecular weight of the preparation in the first line is, of course, smaller than the molecular weight of the preparation in the second line.

You can see that the ethylene oxide with 12 and 3 mols respectively, aims at the same kind of information.

Q. Can you give us a simple definition of what the term "mol" means? A. In chemical reactions, molecules react with each other by numbers.

Let us say a molecule which carries a hydroxyl group reacts with a molecule which carries an isocyanate group.

How heavy the backbone is really makes no difference, it is a question of equal numbers and equal numbers are equal molecules and equal molecules, equal number of molecules is abbreviated by saying equal mols.

(63) Q. Go ahead. A. Then the result of the 2 or 4 reactions really is then described.

First, how do the condensates look and I read now Example 11, line 26:

"The products of these reactions were all clear viscous liquids except the solution of adduct prepared from 3.1 moles of propylene oxide with 1 mol of glycerine, which product was a cloudy, viscous liquid."

In other words, the polypropylene condensate of the lower molecular weight was clouded or cloudier than the others.

Of course, now comes the second step: "On being heated to 150 degrees centigrade for 3 hours, 4-3/4 hours, 7-1/2 hours and 4-3/4 hours, respectively, these reaction products formed gels which on continued heating for a total of 23 hours at the same temperature produced flexible bubble-free resins that decreased in flexibility with decreasing molecular weight of the condensate used in the preparation of the adduct."

So, briefly, to me the result of this experiment is this:

Molecular weight has an influence. If I make the molecular weight too low, then eventually, the (63a) flexibility of the resulting product is reduced. There is no difference disclosed between the two polyethylene runs and the two polypropylene oxide runs.

Should I go on to the next example?

(64) Q. Yes, go ahead. A. Really those two examples, X and XI, they focus attention on the influence of using the two different alkylene oxides which he uses throughout, one in one example and the other in the other example, in this example both. Also to clarify the influence of molecular weight.

The next example, the next four or five really, are devoted to a detailed study of the influence of the plasticizer in respect to the quantity and also in respect to quality.

Example XII contains four runs, four individual runs where the same condensate of alkylene oxide, in this case propylene oxide and glycerol, was reacted with the

same amount of TDI but in the presence of different amounts of the plasticizer, in this case chlorinated biphenyl. The amount of this component was varied from 80 weight per cent down to 20 weight per cent. And the result is summarized at the beginning of the example that the compositions which were studied in this example yielded substantially bubble-free products which varied with a decreasing amount of solvent and in this case he uses again the word solvent for inert component, from a soft jelly to a tough flexible resin.

(65) This is, of course, what one would expect if one has a large amount of plasticizer in a composition of this kind, that it will be softer and if you lessen it it

will be tougher.

Now this example operates with a specific adduct, namely with the adduct obtained from 13.2 mols propylene oxide with one mol glycerol. This is an adduct which had a relatively large amount of propylene oxide added to the three arms of glycerol whereas Example XIII follows up now by using the other adduct which already had been made up, for Example XI and now he uses it for this series of tests. This is the adduct of 3.1 mols of propylene oxide with one mol of glycerol reacted with the same amount of TDI and now in the presence, again, of 80, 65, 35 and 20 weight per cent of chlorinated biphenyl.

The result again is given at the beginning of the example, that is on Column 6, line 58 and 9, one obtains substantially bubble-free, brittle resins which increased in hardness as the amount of solvent, plasticizer, was decreased.

Well, we understand why it's brittle, because the molecular weight is smaller and, of course, we understand why they get harder when the plasticizer is reduced in quantity.

(66) Example XIV is another example which focuses attention on the influence of the plasticizer. This example actually consists of nine independent runs. It uses the higher molecular weight species of the adduct, 13.2 mols of proplyene oxide, one mol glycerine. It uses again TDI.

A variety of chlorinated compounds were used. They are here not identified as solvents or plasticizers but it says in the presence of a sufficient amount of the following chlorinated compounds.

From 1 to 6 the reaction was carried out while the plasticizer was there and from 7 to 9 the condensation product was mixed with similar amounts of the melted chlorinated compound and then reacted with the TDI. In other words, these runs, 7 to 9, actually studied the influence of a different sequence of the reaction steps or of the various steps. What is studied here specifically is indicated in Column 7, line 29, the time required to get the above products and the type of resins formed are indicated in the following table, and then Table 9 presents the results. All of the resins obtained, several of the resins obtained were bubble-free.

Resin 9 is characterized as a brittle resin with a few bubbles and resin 6 is characterized as a soft (67) flexible resin with a few bubbles.

In other words, this example, again, discusses the consequences of using various types of plasticizers, different manners.

(68) By Mr. Kelton:

Q. Would you proceed, Dr. Mark.

As I recall, we last discussed Example XIV. A. Yes. Examples XV and XVI are also devoted to a study of the influence of the plasticizer on a polypropylene oxide adduct of glycerol with the use of m-tolylene

diisocyanate. In other words, additional information is collected about the importance and the action of the plasticizer.

Example XVII still operates with propylene oxide but uses MDI instead of TDI.

In line 15 of Column 8 MDI is disclosed.

Now, the next examples, XVIII, XIX, XX and XXI,

use ethylene oxide instead of propylene oxide.

The first example operates also in the presence of about 50 weight per cent of a plasticizer. The two next examples, XIX and XX, which also use ethylene oxide in the presence of 50 weight per cent plasticizer are of interest because they are on a larger scale. Particularly (69) Example XX, which starts on line 5 of Column 9, discloses the use of 244 parts of a condensate of ethylene oxide with glycerol and the corresponding amount of TDI.

The large quantity of material obtained, also in the presence of about 50 weight per cent plasticizer, made possible in this case to carry out tests concerning the quality of the product obtained and this was obtained with ethylene oxide and the patentee discloses that a sample of the product of the initial reaction was used to impregnate or laminate the glass case with highly satisfactory results.

Example XXI is another ethylene oxide adduct this time with MDI instead of TDI just in order to establish the usefulness of this other diisocyanate.

That completes all those examples which are listed under the original heading "Manufacture of Substantially Bubble-Free Gels and Resins".

The next four examples are under the heading "Manufacture of Resinous Foams". In other words, another product is now desired and they are characterized by the fact that in all four cases a catalyst was used. In other words, a fourth component was used, as already

indicated in the disclosure earlier, particularly in Column 3, lines (70) 48 to 50.

The first example operates with an adduct described in Example X, which is a propylene oxide glycerine adduct and adds 1 per cent of calcium oxide. The result is a porous resinous foam.

The next example does the same thing only with another catalyst. In this specific case it was lead oxide.

The next example goes back to the castor oil technology that goes back to a trifunctional polyester, polyol to a polyol, triol, and uses as catalyst two organic compounds, which I don't want to describe. They are known catalysts for this reaction. Rubbery foam was obtained again.

The last example repeats Example XXIV only with a castor oil which was pretreated, which was hydrogenated.

All these four examples lead to porous foams, porous materials and employ a catalyst. On top of the other three ingredients, namely, polyol, polyisocyanate and plasticizer.

(71) Q. Dr. Mark, do any of Examples 1 through 21 teach the making of foams? A. No, none of the examples 1 to 21—

Q. That is what I was referring to, the examples. A.—teaches the making of a foam.

Q. I notice following Example 25, there is more of the specification.

Could you briefly comment on it? A. Yes. Following Example 25, the patentee comes back to the use of a non-reactive organic plasticizer, which he has devoted so much work in the examples, and indicates that it would be undesirable to omit this ingredient.

He characterizes it, first, in line 3 of column 10 as a non-reactive organic plasticizer, so this time he uses these words in one sequence. Then specifies again that it should be a high boiler.

Evidently, if it is supposed to be a plasticizer, it should not evaporate during the reaction.

Now, on the average, the temperatures which are used in all the examples during the chemical reaction of the polyol with the polydisocyanate range from 100 to 150 degrees centigrade, if he specifies that it should have a boiling point of 200 to 250, this guarantees it will stay there and this, one might say, is in contradistinction to (72) what one usually calls a solvent.

A solvent is used in order to render a reaction system more mobile and to permit the reacting molecules to collide with each other easier, but after the reaction is over, the solvent in most cases is removed, so the solvent must be a material which hoils at a convenient temperature to be removed.

I think this is essentially what is added in column 10, line 28, in terms of the desirability of the plasticizer.

Then the paragraph from 10, line 35 to 66 comes back

to the catalyst, that is, the fourth component.

If you want to make a foam, then, apparently, a catalyst is either desirable or necessary and a number of catalysts are enumerated here, but, again, I don't think that for the time being, at least at the moment, it is necessary to go into very much more detail.

Q. Would you proceed then? A. At the end just before the claims are formulated, the patentee again comes back to the plasticizer and indicates in more detail that the plasticizer has to be added early, had to be intimately associated with the reacting mixture when the material is finally polymerized to a higher molecular weight.

This is taught and explained in column 11, line 6 (73) to 17, and that is the end of the disclosure.

Q. I am not sure that I got those lines in column 11 that you spoke about. A. Line 6 to 17.

- Q. Do you find the patent in its specification describes any advantage of condensate of ethylene oxide or propylene oxide as compared with each other with the polyhydroxy alcohols for elastomeric properties? A. No.
- (74) Q. Can you tell me whether any claim or any example of the patent shows omission of a plasticizer? A. No.
- Q. I apologize for the way I asked the question. It might be interpreted that your answer was that no, you couldn't tell me, so let me ask the question in these terms:

Does any example of the Heiss patent in suit show any omission of a plasticizer? A. No.

Q. Dr. Mark, does the specification of the patent state any advantage of ethylene oxide or propylene oxide for this reaction with polyhydroxide alcohols? A. The word "advantage" in this case can be interpreted as being advantageous for the course of the reaction or being advantageous for the properties of the final product, so maybe I should answer it in both cases.

As far as an advantageous influence on the reaction itself, that is, speed, smoothness, easy controllability, there is no teaching in the patent.

As far as advantage, at least in one specific application of the final products, Example 20, which uses ethylene oxide and which produced enough material to carry out tests, is reported to have given highly satisfactory results and it is the only instance in any of the examples (75) that such a quality mark would have been expressed.

- Q. Have you read the claims of the patent, Dr. Mark? A. Yes.
- Q. Do the claims specify a polyol and a polyisocyanate? A. Yes, the claims specify a polyol and an organic polyisocyanate.
 - Q. Do the claims specify a plasticizer? A. No.

- Q. None of them do? A. None of them do.
- Q. Do the claims specify a catalyst? A. No.

(76) By Mr. Kelton:

Q. Having had that preliminary discussion between Mr. Sweeney and the Court, that is, Dr. Mark, let me ask you, in Claim 3 what is the polyol? A. The polyol in Claim 3 is a condensate of propylene oxide and a polyhydric alcohol having from 3 to 4 hydroxyl groups.

Q. In other words, it is that portion of Claim 3 which

is set off as Item (1)? A. That's correct.

Q. What is the polyisocyanate? A. Claim 3 only demands that it should be an organic (77) polyisocyanate which means it should be an isocyanate with two or more isocyanate groups.

Q. And what is stated as to the ratio of polyisocyanate to polyol? A. The claim demands that there should be an excess of the isocyanate over that theoretically required to react with all active hydrogens of the condensate.

Q. Was this excess, was that the type of thing that you demonstrated with the beads this morning on a 5 to 4 ratio, or whatever it happened to be? A. Yes, that's correct. It is a prescription or it is a restriction so that one wants to have the average, at least all end groups, to be isocyanate end groups and eventually in excess of isocyanate above that.

(79) Q. Dr. Mark, are you familiar with the Windemuth (80) patent? A. Yes.

Mr. Kelton: I might remark, your Honor, that for sake of brevity I will refer to it as the Windemuth patent. It was issued on a joint application of three different people but I see no reason to use anything except the short term.

The Court: All right.

Q. Can you tell us briefly, Dr. Mark, what the Windemuth patent discloses? A. It deals with high molecular weight polyether urethane polymers.

Q. To the extent possible, Doctor, so we could sort of follow you, where you want to make a reference to

column or line, that would be fine.

I am sorry I interrupted you. A. Then it goes on that polyglycol ethers—I am now starting at Column 1, line 20, not really reading it but more or less commenting on it—polyglycol ethers are produced by polymerizing alkylene oxides, for instance, ethylene oxide or by chemically adding alkylene oxides to mono or polyfunctional alcohols and then he mentioned alkylene glycol which we had already as a difunctional and the pentaerythritol which we had before as a tetrafunctional.

(81) By the way, here I might say that the patent really does not use the correct English expression for this compound which is pentaerythritol instead of pentaerythrite.

Q. Can you tell me whether or not it means the same thing? A. It is the same molecule and it is a tetrol.

It's a tetrafunctional alcohol.

Then these materials, these polyglycol ethers which can be either difunctional or trifunctional or tetrafunctional, they must have at least two terminal hydroxyl groups and they are then reacted with isocyanates and in Column 1, 46 and 47, the patent mentions the same two disocyanates which are also mentioned in Heiss and which we have abbreviated as TDI and MDI.

The next teaching of the patent is that if one uses difunctional agents, one gets linear products which are thermoplastic. That is explained in Column 2, line 3 to 8. They are thermoplastic, soluble and they can be used for a number of purposes.

Then he explains that as soon as one uses three or more ols in the polyol component, triols and tetrols, then one gets cross-linking and eventually reticulation (82) or network formation depending on the degree of polymeriz ion of the polyol, that is in reality depending on the lengths of the chain between the cross-links, the products are soft and rubbery or when the network is very dense the material becomes eventually hard.

The next important teaching is that—and that's in Column 2, line 59, that important products are obtained as soon as one uses an excess of the isocyanate over the hydroxyl groups of the polyol. This excess has the same consequences which it has in the Heiss patent or in the Heiss disclosure, namely to have terminal isocyanate

groups which can undergo additional reactions.

If I may summarize the teaching until now, the patent teaches alkylene oxide condensates of diol, triols and tetrols. It teaches the reaction with polyisocyanates and it teaches an excess of the isocyanate groups over the hydroxyl groups. Those are the three essential teachings of that patent.

- (83) Q. Doctor, let me ask you whether to a chemist Windemuth does or does not disclose a condensate of propylene oxide and a polyhydric alcohol having three to four hydroxyl groups? A. Well, the teaching of the patent discloses it because of the fact that alkylene oxides, as they are mentioned already in Column 1, 22 and particularly then in 23, in the plural, embraces ethylene oxide and propylene oxide and maybe one or two more alkylene oxides. This is what, according to his patent, is supposed to be reacted with trimethylolpropane, which is a triol.
- Q. To a chemist does Windemuth disclose an organic polyisocyanate? A. Sure.
- Q. To a chemist does Windemuth disclose reacting such a condensate, as I referred to in my previous question

once before, with an organic polyisocyanate wherein the polyisocyanate is in excess of that theoretically required to react with the reactive hydrogens of the condensate? A. Yes. Specifically, he discloses it in Column 2, lines 59 until 63.

Q. Dr. Mark, with regard to Windemuth, the Windemuth patent, I mean, a statement has been made and I quote:

"Windemuth discloses only ethylene oxide."

(84) As of 1950 and as a chemist can you tell me whether that was correct or incorrect? A. I think that was incorrect.

Q. Now, do I correctly understand from your testimony that the term alkylene oxides has for a long time been used to include to the knowledge of chemists ethylene oxide and propylene oxides? A. Yes.

Q. With regard, Doctor, to your answer to a previous question and my question was with regard to the statement:

"Windemuth discloses only ethylene oxide", and you said I was incorrect, may I ask you why it was incorrect? A. Windemuth in Column 1, line 21 specifically says:

"By polymerizing alkylene oxides"—plural—"for instance, ethylene oxide or by chemical addition of alkylene oxides"—again plural—"to mono or polyfunctional alcohols".

Now, somebody tells the chemist, use alkylene oxides. Then he will use ethylene oxide or propylene oxide mainly, maybe one or two others, if they are available, and if they existed at that time, and I think a (85) very clear illustration of this point of view is the fact that Heiss himself in his own patent about the same time in Example XI teaches the following and I quote from line 11. Column 6:

"Meta-tolylene diisocyanate containing about .23 per cent of hydrolyzable chlorine was reacted with the following alkylene oxide condensates alkylene oxide condensates of gylcerine . . ."

Now, the condensate is in the plural so it would have to do with several alkylene oxide condensates; more than one.

Then, there are four. Two of them are made with propylene oxide and two of them are made with ethylene oxide, which I cannot explain in a different manner than that the word alkylene oxides refers to both ethylene oxide and propylene oxide. That was the usual way of expressing materials in compounds of this kind. Otherwise Heiss wouldn't have done it.

- Q. Dr. Mark, you have said that that was common at the time. Do you have any illustration in the art of its use? A. In fact, there are many instances I know of and probably there could be many more collected, but one—
- Q. I show you United States Patent 2,527,970, (86) patented October 31, 1950, in the name of Herman Sokol, assignor to Hayden Chemical Corporation, and ask you if that is one of them? A. Yes. This is one of the documents which illustrate the use of an alkylene oxide comprising propylene and ethylene oxide.

Mr. Kelton: I would ask that the Sokol Patent, No. 2,527,970, be marked for identification as Plaintiff's Exhibit 6.

Mr. Sweeney: No objection, your Honor.

Mr. Kelton: In view of the fact, your Honor, Mr. Sweeney says he has no objection to marking it for identification, I will hereby offer it in evidence. I intended to anyway, but I didn't want to get into a procedural hassle.

Mr. Sweeney: I am sorry, I thought you had offered it. I don't object to your offering it either.

The Court: It will be received.

(Plaintiff's Exhibit 6 was received in evidence.)

(87) Q. Where does the Sokol patent show this, Doctor? A. The Sokol patent in column 1, line 4, talks about "alkene (EP) epoxides, alkene oxides."

Now, this is, linguistically, not the same as alkylene oxide, but, in fact, it is the same. It is a kind of an abbreviated expression for alkylene oxides, so instead of alkylene oxide, one has said alkene oxide. Instead of propylene oxide, one has said propene oxide. Those are the same thing.

Just in order to illustrate that, I would like to draw your attention to Example 23 of the Heiss patent where in column 9, line 46, he also makes use of this abbreviation. Throughout the patent, he has always said propylene oxide and here he says propene oxide. That is column 9 of the Heiss patent, line 46.

There is nothing wrong with that. I just wanted to eliminate any doubt that an alkene oxide is what we are here calling alkylene oxide.

Now, then, with this understanding, the Sokol patent mentions specifically propylene oxide in column 1, line 9, as one of the alkylene oxides.

He mentions specifically ethylene oxide in column 2, line 2. This is now the Sokol patent.

A few lines lower down, he actually gives the (88) formula of ethylene oxide, the same formula which we had here on one of the charts.

In column 3, line 29, he discloses that propylene oxide and similar alkene oxides, that shows there are other alkene oxides than propylene oxide, for instance, ethylene oxide.

Farther down in column 3, Example 1, deals with propylene oxide, as the title indicates.

Farther down in column 4, line 31, again propylene oxide is mentioned as a reactant.

Farther down, column 4, line 72, ethylene oxide is mentioned.

So this disclosure shows that similar alkene oxides are propylene and ethylene and he actually mentions another one in Example 4, which is column 6, Line 17 or 18, butadiene monoxide.

That is another alkylene oxide which plays no role in our other discussions.

But Sokol patent reconfirms the position that alkylene oxide comprises ethylene oxide and propylene oxide and maybe one or two more.

(97) By Mr. Kelton:

Q. Dr. Mark, I show you Plaintiff's Exhibit 7-A and I show you a paper having in the upper right-hand corner the handwritten letters M-106 and entitled "Industrial Application of Diisocyanate," and ask you whether this paper to which I have referred to is or is not a translation of Exhibit 7-A?

Let me ask you first have you had any chance previously to compare this paper marked in the upper right-hand corner M-106 with Plaintiff's Exhibit 7-A? A. I know the German text since many years. I have obtained the translation only about six weeks ago but I compared the translation with the German text and with the exception of a few minor, I would say, slips, I would say it is a correct and true translation.

When I say minor slips, for instance, the termination reaction is called the chain breaking reaction. It is not exactly what we would say, but it means the same thing.

In most instances, it is a perfectly acceptable translation.

(98) By Mr. Kelton:

- Q. Dr. Marks, do you have a copy of Plaintiff's Exhibit 7-B for identification, that is, the translation? A. Yes, I have.
- Q. Who is set forth as the author of the article of which this is a translation? A. Dr. Otto Bayer.
- Q. Are you acquainted with Dr. Otto Bayer? A. Yes, I know Dr. Bayer since forty years and, in fact, we are very close friends.
- Q. Does Dr. Bayer have any particular place in polyurethane chemistry? A. He is the originator of it. I think in polyurethane chemistry Dr. Bayer has played the same role (99) which Dr. Carothers of DuPont has played in the chemistry of polyamides and polyesters.

Both have been the originators of a new branch of polymer chemistry.

- Q. Do you find any portion of the article with reference to the translation, do you find any points in this rather voluminous document, the translation, that you consider are particularly pertinent as art prior to Heiss?
- (102) I think the most important contribution of this article is a clear establishment of the principles. In fact, it was the first establishment of these principles. If I may refer you to page 5 of the article and if I may ask for the little chart I have made—then at the bottom of this page there appear these two lines which illustrate the reaction of a diffunctional glycol with a diffunctional isocyanate and the arrows indicate how the hydrogen from the hydroxyl group migrates to the nitrogen and how finally a linear polyisocyanate results.

On one of our charts we had this same scheme but this was a copy of the original elucidation of this reaction by Dr. Bayer. That is the first thing.

Mr. Kelton: May I ask that a copy of the chart which Dr. Mark has just used be marked for identification as Plaintiff's Exhibit 8-A.

(Plaintiff's Exhibit 8-A was marked for identification.)

The Witness: The next point is to be seen on page 6 in Fig. 1-A of which I also have made an enlargement and it refers—well, there the linear polyicocyanate of (103) the preceding page is now actually depicted as it would look molecularly with the individual atoms and with the bonds between them. So this is a linear polyisocyanate chain produced by the interaction of a difunctional polyol and a difunctional diisocyanate.

The important principle which again was established at that time and, in fact, in this original figure for the first time—now we know it all, of course—is that if one applies at certain points trifunctional glycols or trifunctional polyols like glycerol then one gets a network which may be elastic, may be hard, depending upon what is between the fixed points of this network. This was the second important fundamental knowledge which Dr. Bayer established while he worked in the field.

There exists linear polyurethane if one operates exclusively with diffunctional ingredients and one always gets networks if one introduces tri or tetrafunctional ingredients. This is then demonstrated in the paper in many examples but unless it is demanded. I think the important thing is the establishment of the principle.

A third interesting contribution of the article in view of the Heiss patent is contained on Table 2 on page 16. This table has to do with linear polyisocyanates because it operates only with difunctional (104) isocyanates and

difunctional glycols. It enumerates a large number of glycols. In fact, many of them appear many, many years later in the Heiss patent.

Of special interest is an ether glycol or two ether glycols. They are down the line number 8 and 9.

Q. Are the ones in the next column opposite an FP-120 and 124? A. That is correct. They have fusion points. FP means fusion points. In other words, they have melting points of 120 and 121.

Why are they interesting? Because they show that other glycols can be used and have been used because if they wouldn't have been used they couldn't know the melting points. Those products have been actually prepared and studied.

Still a little bit farther down where a melting point of 208 to 212 is indicated we even have a polyether polyglycol.

A polyether polyglycol is a material which is mentioned in the Heiss patent and actually this specific polyether polyglycol figures as an example in a German and in a U.S. patent.

It figures as an example in a patent and was actually prepared somewhere around 1937 or 1938. We might (105) talk about that later.

In other words, this material is a polyether polyol and together with the disocyanate gives a linear polyother urethane or polyether polyurethane. It just shows that components have been used at that time which contained ether bonds.

Q. Doctor, before you leave that, let me ask you—because this much hasn't been translated—will you tell me what these headings mean in English? A. Well, the headings in English—the left heading is diisocyanate and the next means glycol which is also the same word.

The next means FP, that is point of point of fusion, the melting point, and the last means properties, that is a German word.

Q. In the column properties with reference to the two ether glycols to which you referred there is some statement in the properties column and what does it say? A. The statement says that it is very ready to be converted in a fiber, being linear, of course, it's a fiber forming polymer.

Q. With regard to the polyether polyol to which you called our attention which is set forth in a point of fusion of 208 to 212, what is the ratio over in the (106) properties column? A. The remark in the properties column says that it gives elastic fibers.

The Court: What is the translation of the ether? The Witness: Polyether polyol.

The Court: I have that. That is the elastic fiber. But the one above that that you referred to before?

The Witness: It's just a fiber former but it doesn't give an elastic fiber.

The Court: I see.

Thank you.

Q. I interrupted you to ask you about the untranslated portions of the table, Doctor.

Will you go ahead. I am sorry. A. In the balance of the publication a few other points might be mentioned, particularly on page 26, there is a long table in which a large number of di and triisocyanates are disclosed, and on page 28 where a large number or a considerable number of triol or polyols are disclosed. They are all polyester polyols. They are like the castor oil example in the Heiss patent. They are (107) a polyester polyol.

The teaching of the patent or of the article then specifically goes to the difference between divalent and polyvalent materials.

The polyvalent materials are cross-linked networks and the divalent materials are linear. This has already been indicated before but there exists a number of examples for that.

The article also discusses in detail or in some detail the problem of ratios of the reactive groups and, of course, indicates that if you want at the end an excess of hydroxyl groups you have to have them in excess over the diisocyanate or polyisocyanate groups. If you want to have at the end isocyanate groups at the surface of your molecule or at the ends of the chains then the excess has to go in the other direction. So he explains to a chemist what he has to do when he wants to get a certain species of polymeric material.

Under certain conditions he has to use exact equal amounts molecularly speaking and on one occasion he has to go to an excess of hydroxyl and in the other he has to go to an excess of isocyanate and it all, I think, can be summarized in one sentence on page 6 where he says as a result of this work and the considerations—it's in (108) the middle of page 6. He says:

"In fact, it is now possible to construct high molecular compounds like a builder by starting from defined polyoxy compounds of any desirable form without difficulties."

I think this sentence really wraps it up and then the rest of the article is just a justification of the sentence.

Mr. Kelton: I request that the second chart to which Dr. Mark referred, the one showing the linear elastic structure and the elastic retriculated or net structure be marked for identification as Plaintiff's Exhibit 8-B.

(Plaintiff's Exhibit 8-B was marked for identification.)

Mr. Kelton: I offer into evidence as Plaintiff's Exhibit 9 the United States Patent No. 2,726,219

to Frederick B. Hill as assignor to E. I. DuPont de Nemours & Company.

Mr. Sweeney: No objection, Your Honor.

(Plaintiff's Exhibit 9 was received in evidence.) (109) Q. Dr. Mark, before we leave this translation, Plaintiff's Exhibit 7-B, let me ask you what types of backbones go to make up these building blocks as Bayer shows? A. Bayer shows that many kinds of different polyesters, many kinds of different glycols and in that table, too, there were about 25 of all kinds of different types of backbones between the two functional groups. They all can be used. They all have been used. They all work.

So do the polyesters which are on page 29 of the translation in the table of the Desmophens, many different types of combinations, and they all work, so whatever the backbone is, if one has diffunctional reactants, one gets linearity, and if one has polyfunctional reactants, i. e., three or four functional materials, one gets networks.

Could we turn now, Doctor, to Plaintiff's Exhibit 9, the Hill patent.

Mr. Kelton: I might remark to the Court that the Hill patent was issued on an application filed August 24th, 1951. Under the provisions (our laws interpreted by the courts it, therefore, has a prior art date, having issued as a patent it has a prior art effective date as of its filing. For that I would refer (110) your Honor to page 12 of our brief, Footnote 4, which refers to the case of Hazeltine v. Brenner, 382 U.S. 252 in 1965.

Q. Doctor, would you explain the Hill patent to us in connection with its features which you consider relate to various aspects of the Heiss claims? A. The Hill patent teaches the production of polyurethanes by the interaction, by the chemical reaction of polyalkylene, ether

glycols with alkylene diisocyanates, which is spelled out in Column 1, line 52. It has already been said that polyalkylene ether glycols are bifunctional because they are a glycol.

The alkylene residue may be either ethylene or propylene and, in fact, in the examples Hill shows and teaches in Example 1 the reaction of a polyethylene ether glycol, that is in Column 3, line 15, a polyethylene ether glycol with TDI, with tolylene isocyanate in Example II, line 40 of Column 3, the reaction of a polypropylene glycol in this case with MDI or with TDI.

So, we have the element of a linear polyalkylene ether glycol, ethylene or propylene, reacted with a polyisocyanate.

The patent also teaches an excess of the isocyanate group over the hydroxyl group. This is spelled out specifically in Column 2 in lines 53 down to (111) 65. So those are the three important elements.

Q. Now, in connection with lines 63 to 65, I notice that with regard to the ratio of disocynate to polyethylene glycol in line 59, it says:

"The weight ratio." A. Yes.

- Q. Will be and so forth. Is that the part that you referred to for demonstrating the excess or do you prefer to refer to provided the mol ratio is between 7.5 to 1 and 12.0 to 1? A. This is the important point in comparison with the Heiss patent because the excess in patent refers to the mol ratio. So here the mol ratio between 7.5 to 1 and 12 to 1 in one case and 1.5 to 1 and 4.5 to 1 in the other case.
- Q. When you say mol ratio, may I ask you mol ratio of what? A. Of the isocyanate groups to the hydroxyl groups.
- Q. Doctor, I would like you to compare, if you will, the disclosure of Hill, on the one hand, and the subject matter defined in Claim 3 of the Heiss patent, on the other hand.

If you have those two before you, I will ask (112) you whether there is any difference between the disclosure of Hill and the subject matter defined in Claim 3 of the Heiss patent? A. Yes.

Q. What is it? A. The difference is that Hill operates with difunctional polyalkylene, ether polyethylene or polypropylene, ether glycols, whereas in Claim 3 it is said that the reaction product of a condensate of propylene oxide and a polyhydroxy alcohol having from 3 to 4 hydroxyl groups.

Q. In other words, if I understand you correctly, you are saying that the only difference is that Hill uses a dihydric alcohol or diol and Claim 3 of the Hill patent specifies a trihydric alcohol or triol? A. And tetrahydric alcohol or tetrol.

Q. And tetrahydric alcohol or tetrol? A. Yes; that is correct.

(123) Q. Dr. Mark, as to the group of charts, a copy of which are marked for identification as Plaintiff's Exhibits 4-A to 4-L. I think you have not made any comment on 4-F. Can you just very briefly tell us what it shows? A. I have not made any comment on 4-F up to now really as its content was not needed and, in fact, it may not be needed in the future either.

What it illustrates is the reaction product obtained if ethyleneglycol, which is here in the middle of the chain, if this is reacted with a mixture of ethylene oxide and propylene oxide so that the chains, which grow out of each of the reactive hydroxyl groups, each of the functional groups, are actually a mixture of the two monomeric units employed.

Materials of this type are called co-polymers and in certain examples of some of the patent reference is made to such co-polymers. That was the only reason why

I thought we might like to have this chart maybe later for reference. It wasn't discussed yet.

(125) Q. Dr. Mark, yesterday afternoon near the close of the session we were discussing the Hill patent. As I recall, you discussed the points that you felt were significant in that Hill disclosure.

Do you recall that? A. Yes.

Q. Let me ask you: In the light of the art prior to Hill would or would it not be apparent to a chemist to employ Hill's polypropylene ether backbone in a cross linked or network polymer? A. Maybe I might point out that the prior art, which (126) I would like to use in order to formulate my answer, is represented here (indicating)—

Mr. Kelton: May the record show that the witness is referring to 8-B.

A. (Contg.) Because in this exhibit there is a linear polyester chain obtained by the interaction of difunctional units of glycols and dicarboxylic acids and, as said in the text and as shown here, if one wants to proceed from the preparation of a linear product to a network, one has to introduce trifunctional units in the original chain.

So if somebody has at his disposal a chain made up from di-functional units—in this case ester units; in the Hill case ether units—and then if he wanted to go to a network, evidently what he would do, he would do the same thing that Bayer did much earlier in using trifunctional units in the chain.

In this specific case of Hill, it would probably be glycerol.

Q. Can you tell me how this chemist to whom I referred would be taught to do it? A. What the chemist would do, he would introduce here in huilding up the polyether chain instead of using di-functional units, he would use trifunctional units.

(127) Q. When you say di-functional units instead of tri-functional units, do you mean he would react the propylene oxide with a tri-functional alcohol, in other words, a trihydroxy alcohol instead of with a di-hydroxy alcohol? A. Instead of using some other glycol to build up the polypropylene glycol, he would look at this chart and use a tri-functional—tri-hydric alcohol, for instance, glycerol in order to build up his polypropylene oxide polyol.

Q. What, if anything, would teach him that by using a tri-functional or tri-hydroxy alcohol instead of a difunctional or di-hydroxy alcohol he could achieve this reticulation? A. The teaching for that is whether one gets a linear product or a network doesn't depend on the character of the backbone of the chains. It depends on the polyfunctionality of the used ingredients, the principle which Bayer taught many years ago.

Q. Let me ask you, Dr., whether this transfer from the use of a di-functional material giving a linear material, whether the transfer from that linear material to a cross-linked material is shown in the art prior to Heiss other than in the Bayer reference to which you have referred, Plaintiff's Exhibits 7-A and 7-B? (128) A. Yes.

Q. Where? A. It is shown in an article which appeared in an American journal, namely, known as Chemical Engineering, and it was published as the result of work which was carried out at the University of Princeton.

Q. I show you a photocopy taken from Chemical Engineering and ask you if that is the article you referred to? A. Yes, this is the article which I am referring to, specifically to pages 165 and 166.

Mr. Kelton: I offer in evidence as Plaintiff's Exhibit 10 a photocopy of the cover page, a portion of page 3, which is the masthead page and shows the copyright notice of 1950, and the rest of

page 79 is not important, and pages 165 and 166, all of the April 1950 issue of Chemical Engineering.

Mr. Sweeney: No objection, your Honor.

The Court: Received.

(Plaintiff's Exhibit 10 received in evidence.)

Q. Dr. Mark, will you tell me what kind of a publication Chemical Engineering is? A. Chemical Engineering is, I think, one of the representative publications in this country covering all (129) kinds of articles, reports and events in the entire field of chemical engineering.

Q. What issue of Chemical Engineering is that a copy of? A. This is a copy of the April 1950 issue of Chemical

Engineering.

Q. Would you refer to the matter that you have indicated on pages 165 and 166? A. The title of the article on page 165 is "Princeton Introduces Novel Polyurethanes Made From Castor Oil and Diisocvanate."

The article then describes where it was done, in the plastics laboratory of the University of Princeton, and goes on to indicate—and this is in the second column, about the middle of the second column—that disocyanates react and have been reacted with glycols and, of course, one gets linear chains.

In the third column, the last paragraph has the heading "How They Are Made", and there it says, if I may actually quote:

"What we do is react Castor Oil with a diisocyanate. Castor Oil is basically a trihydroxyl alcohol and it forms cross linkages with diisocyanates."

Actually, what has been done here in 1950 is very close (130) or is close, I should say, to what has been done in Example 9 by Heiss with the distinction that in the case of Heiss, of course, a plasticizer was present, but the basic reaction and the principle of obtaining cross linked

ether diisocyanate reaction products is described here by employing Castor Oil with a diisocyanate.

The Court: May I interrupt with a question? Mr. Kelton: Yes, sir.

The Court: I think I understand, Dr. Mark, that you have indicated that Bayer had shown that by using a glycol mixed with an alkylene oxide that you got the linear line and that if he mixed it with what you call a tri-functional glycerol with the alkylene oxide that he got a network.

What I want to know—and I suppose I am ahead of the story somewhat—is why would a chemist—to me all this looks all very esoteric—what is the advantage in terms of development of materials between the cross linkage and the linear?

Maybe I am going ahead, but I would like to get some understanding of that rather than an abstraction.

The Witness: The advantage of having cross linked systems, whether they are polyethers—as we are now discussing—or whether they are polyesters, as originally (131) Bayer did, is that, for instance, if you want to make a foam, the foam of a linear polymer will be thermoplastic, will yield under weights, whereas the foam of a network is something fixed, permanent.

The Court: And solid? The Witness: And solid.

Now, that was nothing new in the art. The same thing is and has been done with rubber for many, many years. Rubber as such is a linear polymer.

Rubber corresponds to the upper part of Exhibit 8-A. But rubber alone is no good because it eventually yields and gives a permanent set. Therefore, one has to vulcanize rubber. That was the important and crucial discovery of Charles Goodyear a hundred years ago and the

vulcanization of rubber is exactly the same reticulation by another chemical reaction, but the principle is the same. Out of the thermoplastic they make a termoset material which does not yield and creep any more. That is the important thing.

The Court: Now, at least, I have some feeling about

this. Thank you.

By Mr. Kelton:

Q. Dr. Mark, in your comments you used the term thermoplastic and thermoset. I wonder if you could tell us what you mean by those? (132) A. There are two large classes of plastics. Those that can be reversibly softened and hardened. They are called thermoplastic. They are very useful because you can mold them. At an elevated temperature you mold a cup and then you cool it down and the cup is hard, but you can remold the cup, if you want to, or you can remold the waste which results during the fabrication of the cup.

All themoplastic materials belong to the linear group.

On the other hand, however, sometimes you want something which does not need to be softened. For instance, a telephone case or a radio or a television case. There you want it to be like wood, hard forever, not fuseable and not meltable; just like wood.

These materials are cross linked, as is wood, and they are called thermosetting.

Q. As I understand your discussion of the article, which is Plaintiff's Exhibit 10, it involves the reaction of Castor Oil with disocyanates? A. That is correct.

Q. Would you refer, Dr., to Example 9 of the Heiss patent and tell me if there is any relation between that example and the April 1950 article, which is Plaintiff's Exhibit 10? A. Example 9 appears in the Heiss patent in column (133) 5, line 65, and it teaches the reaction of Castor Oil with a diisocyanate, specifically TDI, in

the presence of a plasticizer. The plasticizer is not partaking in the chemical reaction, which is specified in the patent by saying the plasticizer must be a non reactive organic liquid or solid, so the chemical reaction takes place exclusively between the Castor Oil and the diisocyanate exactly as taught in the article in 1950.

Q. Dr., with regard to Plaintiff's Exhibit 10, what does it show, if anything, with regard to ratios of isocyanate groups to hydroxyl groups? A. At the bottom of the third column on page 165 and on the top of the middle column on page 166 the article spells out the quantities of the reagents involved and this quantity indicates an excess of isocyanate groups over hydroxyl groups.

Mr. Kelton: I offer in evidence as Plaintiff's Exhibit 11 a copy of British patent specification No. 793,780.

The Court: Mr. Sweeney?

Mr. Sweeney: Your Honor, I am looking for that exhibit. Was I given notice that you intended to use this exhibit?

Mr. Kelton: This is not prior art; it is not (134) presented as prior art.

Mr. Blecker: Yes, Mr. Sweeney, you were given notice. Mr. Stapleton of your office was advised of both this British patent and the Australian patent; that they would be offered into evidence.

Mr. Sweeney: May T ask then what the purpose of the exhibit is, if it isn't prior art?

Mr. Kelton: Yes, I will be glad to explain.

If the Court please, this and the next exhibit will be offered and are, respectively, the British and Australian counterparts of the Heiss application.

For example, in this British specification, which issued on the specification filed, it says:

"Application made in the United States of America on August 7, 1953."

Its subject matter is substantially the same, that is, the filing date of the Heiss application. In other words, under the International Convention, Heiss and Saunders, as assignors to Montsanto Chemical Company at that time, had advantage as a filing date of the U.S. Filing date so far as prior art in England went and they treat this under the International Convention because it was with the same disclosure.

Now, as to relevance, the deposition testimony that (135) we will read and exhibits which were developed in the deposition, we will show that in the UK, that is England, let's say, and in Australia, the applications were filed and unamendable before the Price patent issued and became known to Heiss and his people.

Therefore, I think we will convince your Honor that all of the claims of these two foreign patents do call for a plasticizer. It is evidence that not until Price did Heiss become aware of anything that he could regard as his invention without a plasticizer.

The Court: Right.

Mr. Kelton: There are comments and letters, Mr. Upchurch in one letter indicates that it is with regard to the British and Australian patents, it is too late to change those. That is the relevance.

The Court: I understand the point.

Mr. Sweeney: Your Honor, insofar as it affects the patent as it was issued, there is no question about that. I question the relevance myself.

In the first place, it is a patent issued under a different patent system, under different patent laws, so what was issued there really has no bearing on what is issued here.

Furthermore, the point that Mr. Kelton apparently is (135A) headed for and has been headed for for two days was decided by the United States Patent Office adversely to General Tire during the Price-Heiss interference, so this in my judgment is burdening the record unnecessarily with documentation that has no relevance to this case.

(136) Court: Well, I think that when all the evidence is in, I'll be able to decide that but, at this point, I will admit the document.

(Plaintiff's Exhibit 11 received in evidence.)

Mr. Kelton: Now, Mr. Sweeney, could we stipulate that Plaintiff's Exhibit 11 is issued on an application which is the British counterpart of the United States Patent application 373,036?

Mr. Sweeney: Is that the Heiss US application? Mr. Kelton: Yes. it is.

Mr. Sweeney: That statement connotes a lot of things and I am not so sure I want to stipulate to it. The British counterpart of—what do you mean by that? Do you mean that this is the British application that was filed based on the US case? If so, I'll agree to that.

Mr. Kelton: All right. I'll accept that concession.

I offer in evidence as Plaintiff's Exhibit 12 an Australian Patent, number 205,456, issued to Monsanto Chemical Company, as applicant, and noting the actual inventors to be James Henry Saunders and Herbert Lewis Heiss.

The Court: I will admit it. I understand, of course, Mr. Sweeney, that you make the same objection as to Exhibit 11.

Mr. Sweeney: Yes, your Honor: same objection. (137) (Plaintiff's Exhibit 12 received in evidence.)

Mr. Sweeney: My objection is preserved, your Honor.

The Court: Yes. I just stated that.

Mr. Kelton: Mr. Sweeney, may we have the same concession as to Plaintiff's Exhibit 12 as on the application based on the United States Heiss application, serial number 373,036?

Mr. Sweeney: I will agree that the Australian application was based on the US application.

By Mr. Kelton:

Q. Now, Dr. Mark, I show you Plaintiff's Exhibit 11, which is a British patent, and ask you whether or not all claims specify a plasticizer.

Mr. Sweeney: Your Honor, I'll object to this line of questioning. The documents speak for themselves.

The Court: It's overruled. You can answer it.

A. Yes, they all do.

Q. I show you Plaintiff's Exhibit 12, an Australian patent, and ask you whether or not all of the claims specify a plasticizer. A. They do specify a plasticizer.

The Court: Is the plasticizer the nonreactive organic liquid?

The Witness: Yes, your Honor. In the disclosure (138) of the patent, it is said that the plasticizer is a nonreactive organic liquid and in the claims individually it is spelled out that the reaction should be carried out in the presence of a nonreactive organic liquid.

Q. Dr. Mark, you answered with regard to Exhibit 11, the British patent.

Would you just briefly point out where in the claims a plasticizer is called for? A. On page 9 of Exhibit 11, Claim 1, line 15, spells out that the reaction should be

carried out in the presence of a nonreactive organic compound.

Q. Well, doctor, how in the claim language can you tell that is a plasticizer, rather than a fairly low boiling solvent? A. Because in the same claim at the beginning, that is in line 9 and 10, it is said "a process for the production of a substantially bubble free plasticized urethane or thiourethane condensation polymer."

Mr. Kelton: As to Claim 2, your Honor, I can tell you that, since it relies on Claim 1, is dependent on Claim 1, it incorporates all of the limitations of Claim 1, plus whatever is added in Claim 2.

Claim 3 is a process according to either of the preceding claims, so that incorporates Claim 1. Claim 4 is (139) a process according to Claim 3 which, in turn, incorporates the limitations of Claim 1.

Claim 5 is a process according to any of the preceding claims and then deals with the ratio of isocyanate to reactive hydrogen atoms, so that, again, incorporates the Claim 1.

Q. Now, Dr. Mark, how about Claim 6? A. Claim 6, line 44 and 45—

The Court: I can see 6, 7 and 8 specifically mention it, the plasticizer.

Mr. Kelter: All right.

- Q. Now, the Australian patent, doctor. Where in Claim 1 is the plasticizer? A. Claim 1 in the last three lines says, "is carried out in the presence of a nonreactive organic liquid which boils at a temperature not less than about 200 degrees centigrade."
 - Q. Does that define a plasticizer? A. Yes.
 - Q. To a chemist. A. Yes.

Claim 2, the last two lines, "the presence of a non-reactive organic liquid having a boiling point of at least 200 degrees centigrade at atmospheric pressure." (140) Claim 3 refers to the other claims.

Claim 4, the second and third lines, "in the presence of a nonreactive organic liquid boiling at a temperature of at least 200 degrees centigrade."

Claim 5, line second and third, "in the presence of a nonreactive organic liquid boiling at a temperature of at least 200 degrees centigrade at atmospheric pressure."

Claim 6 is referring back to the earlier claims.

Q. Now, doctor, I show you a photocopy of the cover page and pages 72 and 150 of "Chemical and Engineering News," October 14, 1957, and ask you what the material on the righthand column of page 72 is.

Mr. Sweeney: Your Honor, before the witness answers that, I presume Mr. Kelton is going to mark this as an exhibit.

Mr. Kelton: Yes, I am.

Mr. Sweeney: Because I have an objection to its use. May I be heard?

The Court: Of course.

Mr. Sweeney: This document is dated in 1957 and I don't think any of us are disputing the fact that it was published in 1957. Therefore, it cannot be prior art, as against the Heiss application, which was filed in '53.

Therefore, whatever is said in this article is, I presume, what plaintiff wishes to prove. In other words, he (141) wishes to prove what the article says. Whatever the article says is hearsay because the author of the article is not here; I can't cross examine an article; I don't know if this article tells the truth or not.

Therefore, I object to the use of the exhibit.

Mr. Kelton: I will clarify to your Honor the purposes for which it will be offered. This article in Chemical and Engineering News is offered not to prove the truth of the assertions contained in it but to prove that these statements were made; in other words, merely the publication of the thing.

What they are is announcements and there are several of them. This is only the first one. They are announcements by industrial companies, in this case an announcement by Union Carbide that got into the news, that they were going to offer polypropylene hydroxy alcohol condensates in 1957, before Heiss changed his claims, to the trade, to the polyurethane trade.

Now, it's our position that the availability or the notoriety that came from this publication was one of the factors that triggered Mr. Heiss and the people that were working with him in the patent field—that is, his patent attorney—when these were seen, they changed their claims in the file history, as will appear from the file history that (142) is in evidence.

They put in for the first time alkylene oxide claims and this was the preceding step to their seeing the Price patent and saying, "Oh, my heavens, we have been regarding the wrong thing as our invention."

Therefore, I say Mr. Sweeney's objection to hearsay is pointless here, because we don't intend to use this for the truth, to prove the truth of things that are asserted in it here.

What we intend to use it for is to show that it in fact was published and became a matter fully open to the art.

Dr. Mark has testified that this is one of the very widely read magazines.

Mr. Sweeney: May I be heard again, your Honor?

Mr. Kelton's statement notwithstanding, the article is hearsay and there is no proof that Mr. Heiss ever saw it, nor did anybody else connected with him. The inventor is charged, under the law, with the knowledge of the prior art, whether he has seen it or not so that if a document is published years before your inventive work is done, you are supposed to know it, whether you have ever seen it or not.

Mr. Kelton's position now is that based on this publication which he has no proof that anybody ever saw, anybody connected with the inventor, Mr. Heiss, Mr. Heiss (143) did something.

The exhibit is not tied up at all. It's hearsay as to what it says and there is no proof that anybody connected with the inventor ever saw it.

The Court: May I have a copy of it, please?

Mr. Kelton: May I just remark, your Honor, that it seems to me that Mr. Sweeney's latter remarks are directed to weight and not to admissibility for this limited purpose.

The Court: This is Chemical and Engineering News. The objection is overruled and I'll accept it, admit it for the limited purpose, which is to show that the publication of the article occurred and that's all it can be used for.

Mr. Kelton: I offer in evidence the cover page and pages 72 and 150 of Chemical and Engineering News, the issue of October 14, 1957, as Plaintiff's Exhibit 13.

(Plaintiff's Exhibit 13 received in evidence.)

Mr. Kelton: Now I might explain to your Honor that we have included in Exhibit P-13 the cover page of the magazine and page 72 and our only in-

terest is the fact of publication to the industry, which would be accepted by anybody as an offer, only the righthand column on page 72; that is, "more polyether is coming".

Now, at the bottom of that column, it says "continued (144) on page 150." Well, there is no continuation on page 150 but we have included the 150 so that that will be apparent, that there is no continuation.

For our purposes, whatever is said here in the righthand column on page 72 is ample.

Q. Now, Dr. Mark, will you tell us in just general terms what is set forth in this righthand column on page 72 which forms part of Plaintiff's Exhibit 13?

Mr. Sweeney: Did I understand your ruling to be that this was admitted for the limited purpose to show that the article was published, your Honor? The Court: Yes.

Mr. Sweeney: I don't see where this question is germaine, under the circumstances.

The Court: I agree. I think that, Mr. Kelton, the limited purpose for which you offered it and under which it was accepted was merely to show its publication. I don't think that any further discussion of it in the record would serve any purpose. Then you get into the question of its truth, and so forth.

Mr. Kelton: Our problem, your Honor, with thet view is that this is a publication to chemists. It may require some explanation of the chemistry. All I am saying is that we are not putting this in to prove that Union Carbide (145) was selling in tank cars at 25 cents per pound delivered or that this is one of the dominant polyethers in today's market.

That point is not of interest and it does not involve us or concern us but, for instance, the heading is "more polyether is coming," and then "Carbide"—that's Union Carbide—"heads for more of the polyurethane raw material market with polypropylene oxide-hexane triol adduction".

Now, what is hexane triol? What does this mean to a chemist? I would like to have the opportunity for Dr. Mark to tell me what that means to a chemist.

The Court: I have read the column and it seems to me that I will allow that question. That is the only reference in there that I believe I do not understand, so that you can ask that.

I'll allow you to allow Dr. Mark to enlighten me on what that means.

Q. Dr. Mark, what's a hexane triol? A. Hexane triol is a trifunctional alcohol of the same type as glycerol. It's a glycerol-like product and if it is reacted with propylene oxide, it's like nonlinear polyether polyols.

The Court: That's the announcement of the kind of network composition we have been discussing here?

The Witness: Exactly, your Honor.

(146) Mr. Kelton: I offer in evidence as Plaintiff's Exhibit 14, for the same limited purpose that we have discussed in connection with P-13, the title page and pages 29 and 66 of the December 9, 1957, issue of Chemical and Engineering News.

The Court: Mr. Sweeney.

Mr. Sweeney: Your Honor, my objection would be the same, of course, and I gather, since he has offered it for the same limited purpose, that your ruling would be the same.

But I think I can add to my originial objection that we are now getting cumulative. We are put-

ting the same thing in for the same purpose and I suppose you can put in the entire library of the Chemists' Club at this rate.

Mr. Kelton: These purport, your Honor, to emanate from different companies. One has to do with Wyandot Chemical Company on page 29 and another has to do with Dow Chemical Company on page 66.

The Court: It will be received.

(Plaintiff's Exhibit 14 received in evidence.)

Mr. Kelton: In other words, your Honor, the purpose of putting this one in is to show that the previous one was not merely a shot in the dark and it serves also to show the notoriety that these polypropylene or polyalkylene condensates with triols had at about this time.

- offered for that purpose, I will repeat the objection, because the notoriety of what is in these documents is clearly hearsay. Whether or not anybody knew them is a matter that has not been proved before this Court.
- Q. Dr. Mark, I'll ask you, with regard to Chemical and Engineer News, you already testified with regard to its distribution to some extent and, in view of Mr. Sweeney's remarks in questioning whether anybody ever apparently reads Chemical and Engineering News or parts of it, what would you comment on that? A. First, I would like to say that this Chemical and Engineering News about which we are talking now is not identical with the Chemical Engineering in which the article from the Princeton University work was published.

This is a publication which every member of the American Chemical Society gets automatically and, as a consequence, it is more widely distributed than any journal

for which you have to put in a certain application for a subscription.

The Court: Are you talking now about the Chemical and Engineering News?

The Witness: Your Honor, I am talking now about the Chemical and Engineering News of which two issues have (148) been under discussion.

The Court: I understand.

Mr. Kelton: I would offer in evidence as Plaintiff's Exhibit 15 the cover page and page 71 of Chemical and Engineering News, the issue of December 16, 1957. Page 79, your Honor, concerns the Dow Chemical Company.

Mr. Sweeney: I, of course, have the same objection to this, your Honor.

The Court: Of course.

Mr. Sweeney: Again, we are getting cumulative. (Plaintiff Exhibit 15 received in evidence.)

Q. Dr. Mark, you testified with regard to the Windemuth patent in relation to Claim 3 of the Heiss patent. Would you please comment on the relationship of your testimony with regard to Windemuth? In other words, comment on the disclosure of Windemuth, as to the remaining issue claims of the Heiss patent.

Those issue claims are claims 4 to 6 and 8 to 11. A. The claims 4 to 6 and 8 to 11 incorporate certain minor modifications of Claim 3. The relationship between my testimony concerning the Windemuth patent to claim 3 is the same for the other claims.

As an example, I might just mention that Claim 4 in line 47 specifies a trihydric alcohol, whereas Claim 3 (149) specifies 3- and 4-hydroxyl groups.

Claim 4 also, in line 49 and 50, 51, specifies that the reaction should be carried out until a second polymer with

a higher molecular weight is obtained. Well, that is, of course, actually the purpose of the reaction.

Claim 5 introduces, on top of the normal polyisocyanates, polyisothiocyanates. Those are sulfur compounds which correspond to the normal isocyanates. In them, the oxygen of the—NCO group is replaced by sulfur, so they contain the—NCS group.

Q. With regard to Claim 6, that's Claim 5 with an organic polyisocyanate? A. As far as 1 can see, Claim 6 is just identical with Claim 3.

Q. Claim 7 is not in issue. A. Claim 8 has two deviations, if I may say, from Claim 3, in that it restricts the character of the polyhydric alcohol which has either 3 or 4 hydroxyl groups, by demanding that it must have less than 7 carbon atoms.

It also, at the end, specifies that additional polymerization should take place during the reaction.

Claim 9, back to Claim 8-

Q. Could 7 ask you, does glycerol have less than 7 carbon atoms? (150) A. Yes.

Q. It comes within it. A. Claim 9, back to Claim 8.

In Claim 10, glycerin is verbally spelled out as the triol and, again, additional polymerization is incorporated.

Claim 11, finally, introduced again the stipulation that the polyhydric alcohol with 3 or 4 hydroxyl groups ought to have less than 7 carbon atoms and instead of reacting it with a polyisocyanate, it reacts it with an organic disocyanate, which is a special polyisocyanate and again indicates that during the reaction, the molecular weight of the product increases.

Q. Now, Dr. Mark, let me refer to your testimony regarding the Bayer article and the Hill patent and also to your testimony as to what they would mean together to one skilled in the art of chemistry and, with that in mind, let me ask you if your testimony in connection with those matters would apply to issue claims 4 to 6 and 8 to 11 in

the manner of your previous application of that testimony to Claim 3. A. Yes, it would.

(151) Cross Examination by Mr. Sweeney:

- Q. Dr. Mark, I gather from your testimony that you are familiar with the urethane industry. A. I am somewhat familiar with it. Of course, not with all its branches and details.
- Q. Well, urethanes are made today using polyols, isn't that correct? A. That is correct.
- Q. Do you know what the major type of polyol that is used is? A. For the production of polyurethanes?
- Q. Right. A. Polyethylene polyols and polyproplyene polyols.
- Q. Do you know the percentage of polyethylene? A. I don't know the percentage but I presume that polypropylene has a majority, is the larger one.
- Q. Is the most widely used polyol? A. That's correct, yes.
- (152) Q. If I may, I would like to ask you a few questions about your charts. A. Sure.
- Q. Now, Dr. Mark, referring to the propylene glycol, which is shown here, why did you write it in this fashion, with the methyl group up at the top? A. In other words, you ask me why didn't I write the chain of the 3 carbon atoms in the hydroxyl group on each side?
- Q. What I meant was, why didn't you write the propylene backbone, as you have called it from time to time, the 3 carbons in a line? A. Well, you see, because the purpose of this chart was to show how propylene glycol and propylene oxide are related to each other.

If you have the 3 carbon atoms in one row, in other words, if you have not the 1, 2 glycol but the 1, 3 glycol, you can't make propylene oxide. You would get a ring

which has four atoms, 3 carbon atoms and 1 oxygen atom and this is not an alkylene oxide of the type we are discussing here.

Q. But the way you have written the propylene glycol, you have got the hydroxyl groups at the end, haven't you? A. At the end of this, of these 2 carbon atoms, that's correct.

(153) Q. Where in reality this hydroxyl group is a branch off of the second carbon group, isn't it? A. I see. You would like to have the hydroxyl group down here, instead of having it over here (indicating)?

Q. Well, wouldn't that be the common way to write it? A. Well, you see, altogether these presentations are kind of simplifications because between carbon atoms and oxygen atoms, there are valence angles. So, really, one should never write them as a straight line.

Q. But it would appear when you depict the propylene glycol as you have here that the two hydroxyl groups are in the same sort of position, wouldn't it? A. Like here?

Q. Yes. A. Yes.

(154) Q. Well, really, those two hydroxyl groups are different? A. Well, they are different in that one hydroxyl group is in the neighborhood of a methyl group and the other one is not, and that is actually what causes their difference, and this shows this difference.

Q. What do you normally call the hydroxyl group at the right-hand end? A. This is a primary hydroxyl group.

Q. And the hydroxyl group at the left-hand end? A. This is a secondary hydroxyl group.

Q. They are different, aren't they? A. They are different but this formula shows it.

Q. And they act differently? A. They act with different speeds under certain conditions, yes.

Q. Now, if I may have Chart Letter E, I guess that would be 4E.

Now, you have gone through an explanation of the propylene exide and propylene glycol and how you get polypropylene exide.

When we get down to the right-hand portion of the polypropylene oxide molecules, as you have shown it, why do you show methyl groups on the second carbon atom? (155) A. This carbon group is one—this methyl group is one carbon atom distant whereas the other methyl group is next to it.

- Q. The other methyl group is next to it. What I asked is why do you show the right-hand group with the methyl group in the second carbon. A. Because statistically both cases happen.
- Q. You mean statistically it doesn't happen some of the time? A. It does not happen all the time. It happens some of the time.
- Q. In other words, some of the time that methyl group is on the first carbon? A. Like here?
- Q. Yes. A. And some of the time it is on the second carbon.
- Q. Do you know the proportion of when it is on the first and when it is on the second? A. Well, I don't think one could make a positive statement because that depends on the catalysts which were used in the formulation of the polypropylene glycol or polypylene oxide.
- Q. You have no opinion as to what the proportion would be? (156) A. I don't know. I presume that it is probably more often like this.
- Q. So that you say more often this right-hand hydroxyl group is a secondary hydroxyl group? A. That is correct. As it is shown here on this side. You see, that is the reason why on one side I show it this way and on the other side I show it the other way.

Mr. Sweeney: Now, if I might have Chart 4E.

The Court: That is what you have.

Mr. Sweeney: I beg your pardon. I'm sorry.

The Witness: This is 4E.

Q. Looking at the top reaction now, which is propylene oxide, which yields this, as you call it, ether bond, and the dipropylene glycol, why is it here that you have shown one methyl group on the second carbon atom? A. Because the reason is the same. The reason is the same sometimes because the addition takes place, or the ring opening leads to this addition, and sometimes it leads to the addition of the CH₂ group here and the CH₃ group at the end. In other words, to the fact that the terminal hydroxyl group is a secondary hydroxyl group.

Q. And then again, is it true that the proportion is more in favor of the terminal hydroxyl group being a secondary hydroxyl group? (157) A. I would say again the relative proportions depend on the conditions, but in general, this type of formula, or this type of structure

is preferred.

Q. Fine. And then what you have shown in the upper part of Exhibit 4E is a primary hydroxyl group on the right-hand side? A. That is correct.

Mr. Sweeney: If I may now have Chart 4F, please.

Q. Dr. Mark, here you have reacted a mixture of ethylene oxide and propylene oxide? A. That is correct.

Q. Is there a difference in the reaction rates between

ethylene oxide and propylene oxide? A. Yes.

Q. Which reacts first? A. Well, again, it depends on the conditions, but generally the activity between the two components is different.

Q. You have already said that. I want to know- A.

Ethylene oxide reacts faster.

Q. So that you were to react ethylene glycol as you have here with a mixture of ethylene oxide and propylene oxide, which of the oxides would react first? (158) A. The ethylene oxide.

- Q. So that the center of the chain or molecule would be ethylene oxide? A. Well, again, suppose that you have a one-to-one mixture of the two monomers of ethylene oxide and propylene oxide, one will get a preferred reaction of the ethylene oxide and within this preference one will have statistical distribution of the two monomers.
- Q. But you would expect the ethylene would react first? A. Well, I would not say first. It just reacts faster.
- Q. All right. But the way you have shown this molecule, the resulting molecule here, you have a ethylene oxide at the end? A. Yes, and you have a propylene oxide on the other end.
- Q. Isn't it more likely that you would have a propylene oxide on the other end? A. Again that depends on the ratio. I didn't specify that the mixture is a molecular mixture. You see, Mr. Sweeney, what I thought is that these charts should not be exactly representing quantitatively the conditions but that they should illustrate in general terms what happens.
- (159) Q. I see. A. In other words, if you would ask me if I make a publication, if I write a publication, or work out the publication on the copolymerization of ethylene oxide and propylene oxide, I certainly would have to go through all those details.
- Q. What I merely wanted the record to reflect is that these charts really don't depict, at least in the case of this chart, 4F, really don't depict what the resultant molecule would look like. A. They are an illustration of the general principles.
- Q. But normally you would find this methyl group over here on the second carbon atom? A. Yes.
- Q. And normally you would have a secondary hydroxyl group at the end? A. Right here.
 - Q. Now, if I may have Chart G, please.

Incidentally, doctor, would it be fairer to say that the chemistry you have depicted on these charts, to a chemist, is reasonably simple? A. Yes.

- Q. Now, on Chart G we have the depiction that you went through of the reaction between ethylene oxide and (160) glycerine, and then you set set out a formula for that? A. That is correct.
- Q. And then you go to your more or less abbreviated formula where you use ET for the ethylene backbone; is that correct? A. That is correct.
- Q. You then went to, on Chart H, if I may have that one, and you then went to an abbreviated formula for the reaction of glycerine with propylene oxide? A. Yes.
- Q. And you used PR for the propylene backbone, is that correct? A. That is correct.
- Q. Dr. Mark, isn't it a fact that in that particular chemical formulation you would have methyl groups distributed, sticking out from the chain at the propylene site? A. Yes, sure.
- Q. And you don't depict those? A. Well, because the PR includes the methyl group.
- Q. Yes, but isn't it a fact that the methyl group is branched off from the chain very important? A. Well, for all kinds of properties, it is important because—well, I don't think for the purpose of this chart it is important because all that the chart should show (161-162) is if one uses glycerol as the starting molecule for the adduct, then one gets a branched molecule.
- Q. Do I understand you to say that you don't consider the branching important? A. This branching here is very important. And, in fact, in order to illustrate this branching, the chart has been drawn. You are right, that the methyl groups are, as you said, sticking out of the propylene chain, but these methyl groups are never referred to as branches but as substituents.
 - Q. I will use your terminology.

Do you consider the substituents important? A. Sure.

Q. But they are not shown in that chart, are they? A. Well, they are not shown in the sense that PR includes them.

- Q. If one knew the chemistry of the propylene oxide glycerol reaction you mean one would know they were there? A. Yes.
- Q. But for the purposes of an explanation to one who doesn't know they were there, they aren't obvious? A. Well, I mean, C_3H_6 includes the methyl group.
- Q. I think I have your answer; thank you, doctor. A. You are welcome.
- (163) Q. We have a lot of testimony, Dr. Mark, about the subject of excess isocyanate or disocyante. Would you please explain the importance of this excess isocyanate? Why is it important to have it? A. Well, the excess of isocyante groups over the hydroxyl groups is important because once between the hydroxyl groups and the isocyanate groups a polyurethane is formed, whatever excess of isocyanate or disocyanate is present will react towards these urethane groups and produce cross-linking, produce, as it was called in many examples or on many occasions, additional polymerization, additional increase of molecular weight.
- Q. What happens when you make a urethane material and you don't use an excess of isocyanate? A. If you make a polyurethane—

Let us be specific. You take ethylene glycol and react it with ethylene oxide—Sorry.

You take ethylene glycol and react it with a diisocyanate, then you get a linear molecule, and if you have an excess of hydroxyl groups, the terminal groups will be hydroxyl groups. You put in a small excess of isocyanate groups, and the terminal groups will be isocyanate groups but that will be the end of it because there will be no reactive groups left in order to keep on continuing the reaction to a (164) high molecular weight.

Q. Since this particular case is more particularly concerned with propylene oxide reactions— A. The same thing will be true for propylene oxides.

Q. Let us just say that you have a propylene oxide tetrol, and you were going to react that with disocyanate, would you get a product if you did not use an excess of the disocyanate? A. You would get the product but it would be a liquid probably, or it would not be a resinous product. It certainly would not be a material as it is shown in the lower part of Plaintiff's Exhibit—I think it was—S. That is a useful work or a reticulated product.

Q. But I am not sure I understand you, and I want to

be sure I do.

You would get a product which would be different, you mean? A. Yes.

- Q. From the product you get when you used the excess? A. Exactly.
- Q. But you would get some sort of a resinous product? A. Yes. Of course you would get some sort of an adduct.
- Q. What would happen if you ran that same reaction (165) with a strong basic catalyst present such as a sodium? A. Isocyanate?

Q. Yes. A. In addition?

- Q. The isocyanate propylene oxide tetrol reaction, let's say. A. Well, I presume that the presence of a strong base would probably have the consequence that the reaction would be very vigorous.
- Q. But then, addressing yourself to the question of excess or no excess, would you expect to get a product that did not have an excess in that reaction? A. With the strong base present?
- Q. With the strong base present. A. Well, of course, it would be a different reaction. I would not be able to predict exactly what kind of product you would get unless we would have the proportions of the individual ingredients.
- Q. I just thought maybe you could tell me. A. (No response.)

Q. There has been talk also about catalysts: for example, in the Heiss patent there is mention of catalysts of various kinds. In polymerization chemistry is it unusual to use catalysts? (166) A. It is very usual to use catalysts.

Q. And the types of catalyic suggestions that are in the Heiss patent then are relatively common? A. Yes.

- Q. You also, I believe, yesterday, in talking about the Heiss patent, used a three-component system. I think one component was a polyol, one was isocyanate, and you said the third component was the aroclor material or plasticizer? A. Yes.
- Q. The plasticizer does not react at all in the reactions depicted in the Heiss patent, does it? A. Chemically?

Q. Chemically. A. No.

Q. It is really just a solvent? A. Well, I wouldn't call it a solvent because I think the situation might be illustrated as follows:

If one wants to facilitate a chemical reaction between two components, one needs mobility of the molecules in the system. This mobility usually is obtained by adding a liquid.

Now, if that liquid is only added for that purpose, and then removed from the product of the reaction, one refers to this as a solvent or a diluent.

- (167) When, however, the conditions are chosen in such a manner that after the reaction this inert liquid remains in the system for certain practical purposes because of the plasticizer, it in fact—in fact, Mr. Heiss calls it a plasticizer.
- Q. But he also calls it a solvent, doesn't he? A. He also calls it a solvent.
- Q. In the reaction you described in a solvent which is removable, let's say, or let's take benzine, which I suppose would be a classical solvent, the reaction in benzine of

the polyol and the isocyanate in the aroclor material, is it? A. The patent doesn't tell me anything about it.

- Q. I am not asking you what the patent tells you, doctor. You are supposed to be an expert. I want you to tell me. A. Well, with the exception, of course, that at the end you remove the benzine whereas you don't remove the aroclor, or whatever else there is. The reaction as such is the same.
 - Q. Neither solvent plays any reaction role? A. No.
- Q. You heard Mr. Kelton mention the Price patent; Dr. Price's patent? (168) A. Yes.
 - Q. Are you familiar with that? A. Yes.

(169) Cross Examination by Mr. Sweeney (cont'd):

Q. Dr. Mark, would you explain Dr. Price's patent? A. Yes. Well, the Price patent has an objective which is somewhat different; in fact, somewhat narrower than the other patents which have been discussed.

(170) The title already indicates that what the patent intends to accomplish is a polyether-polyurethane rubber. In other words, the patent is directed, or the teaching is directed to how to make a rubber, whereas, in the other cases, many other applications have been visualized: coatings, adhesives, thermoplastic materials, and the like.

The patent then, from column, one line, 16 or 18—the fact that rubbery materials have already been made by the interaction of polyester-polyols with disocyanate or polyisocyanate.

And at the end of this paragraph he says that one objectionable property of the polyester-polyisocyanate products is that their resistance to water is not quite as good as one would like to have it in case of a rubber.

And then he continues that polyethers have a higher stability against hydrolysis, which of course is perfectly

correct, and therefore it might be a good idea to substitute the polyesters by polyethers, whenever one wants to prepare a rubbery material with the aid of diisocyanates.

This is spelled out between lines 29 and 39.

In the next short paragraph from 40 to 45 it then says that one would expect, in this manner, to get new vulcanizable rubbers which have particularly good (171) properties, such as high abrasion resistance and whatnot.

And now comes the actual finding, the invention, namely, that materials of this type can be formed if one reacts alkylene oxide, polyglycol adducts with organic diisocyanates, if they have a sufficiently high molecular weight, and if a trifunctional, or even more functional unit is involved. That is really the content of this important short paragraph between line 48 and line 52.

And then the next paragraph, which goes from column one, line 53 to column 2, line 6, teaches that a certain limiting molecular weight has to be made, or has to be used, and that small amounts of a trihydric or tetrahydric alcohol has to be present, a small percentage or percentages have to be present in the polyether-polyol.

So it spells out the—what are the necessary conditions in order to get a rubber in a qualitative fashion.

Now, then, obviously in Column 2, line 14, beginning at line 14, it then goes on to tell a little bit more in detail what the molecular weight is, and what the amount of branching has to be in order to get products of the desired character.

Then it is explained that in the case of the linear polyglycols, molecular weights as low as 600 are found to give good rubbery products if reacted with a di-isocyanate. (172) This is what holds for the linear intermediates.

If, however, one introduces, as it was taught before, certain percentages, either of a trifunctional or of a tetrafunctional, or even of a more highly functional ma-

terial, that is, if one relies on cross-linking of increased intensity, then the molecular weight of the intermediates becomes, or has to be progressively higher.

Again, a quantitative information is given as to what amount of trimethylolpropane, which is a trihydric alcohol, would require a certain increase of the molecular weight.

Then again, in the next paragraph, the limits, so to speak, between the rubbery materials which one wants to get and between the resinous materials which one doesn't want to get in this specific case, is explained in somewhat more detail. In other words, really, the problem is to have an appropriate balance between molecular weight of the intermediate and cross-linking.

That is the claim in this. It is in two paragraphs. As long as one wants to have an elastomeric, i.e., a rubbery material, one has to maintain a certain balance between the molecular weight of the intermediate and between the degree of cross-linking.

Now, then, the next paragraph refers to an (173) observation, to a discovery, or to a finding, namely, as one builds up the alkylene oxide units, either with a difunctional or with a tri-functional initiator, that propylene oxide units are far superior to those where ethylene oxide has been used. So this is a clear indication that evidently experiments have been made to show that propylene oxide is the choice unit over ethylene oxide.

And the reason is given immediately because the resultant materials have a better water resistance, and also, they have less tendency to crystallize.

Now, practically speaking, that means that whatever you make out of them, rubbers, solid rubbers or foams, would be more flexible and they would be softer than the corresponding products made with the use of ethylene oxide.

And then I am going over now and we are now coming to Column 3 where it is disclosed that one must not use either ethylene or propylene. One can use mixtures. So really the contents of the teaching on Column 3 on top, until line 13, prompted me to make this Chart F, which indicates that in Alkylene oxide condensates one may have more than one unit or one monomer, and it is here. Then later, it is said that the alkylene oxides may be, may eventually result into polypropylene glycols or polybutylene glycols, or, of course, (174) polyethylene glycols, and it is then here indicated that mixtures of them are advantageous or, in any event, can also be used for anybody who wants to practice the invention.

Then he comes back in a more quantitative fashion to the molecular weight limitations in terms of getting a

rubbery product or products.

In the next paragraph, which goes from line 30 to line 40 in Column 3, actually the inventor actually discusses just these copolymers of ethylene oxide and propylene oxide which had been shown in Chart F. Really, this was the reason why the Chart F was made up.

Q. You are referring to Exhibit 4F, just so that the record is clear. A. Yes. Excuse me, please. You are

right. I am referring to Exhibit 4F.

And again, at the end it is said, in this specific case, the poly-alkylene polyol, which is then later reacted, of course, with the di-isocyanate, should have a molecular weight over a thousand.

I think this really concludes the teaching as far as the

polyols go.

Now comes the teaching as far as the polyisocyanates go. Just like in all the other patents, or in any patent, in fact, the individual ingredients have to be disclosed, they (175) have to be described, what can one use if one uses it, how does one use it, and I think at this point it finishes the teaching concerning the composition, mole-

cular weight, and cross-linkability of the polyol component.

Now comes the di-isocyanate component. I don't think that this is particularly—

Q. It is quite similar to what is in the Heiss patent, isn't it? A. Well, it is not only similar to the Heiss patent but it is similar to what Bayer has on page 26 in some table where he has all the desmodur, and of course the two more or less—the two materials which have already been discussed here, namely, TDI and MDI, are included.

Also at the end it is indicated that instead of isocyanates, that is, instead of molecules, or compounds which carry the reactive group NCO, one might also use those which carry reactive group NCS. Those are the thioisocyanates.

Then the next two paragraphs, those which go from 60 to 71, really just show that one must not use the disocyanates in order to react to the polyglycols. One can also use di-carboxylic acids.

Q. I thought I misunderstood you. Do you mean that you don't have to use the di-isocyanate; you can use the di-carboxylic acids? (176) A. Yes, I can use any ester forming di-carboxylic acid.

Q. Which would get you a polyester material? A. This would, of course, go back into the classical polyester rubbers of Bayer, which, however, then, in this case, would contain also important polyether components.

You see, the difference between this disclosure and Bayer is that Bayer used throughout ester bonds and only in a few specific cases he puts ether bonds in his network.

Here one has already a polyether with terminal hydroxyl groups and one now uses dicarboxylic acid and one uses the dicarboxylic acid only to link the terminal hydroxyl groups together and still maintains the backbone of the molecule ether bonds.

Q. All right. Go ahead, doctor. A. All right. Thank you.

Then a few words are said about how the reaction occurs, namely, rapidly at room temperature, and then it is said up there how one eventually can mitigate the rate of reaction so that it would not become too vigorous by actually using not a di-isocyanate but a complex of two di-isocyanates which first have to open up in order to become reactive.

Actually, this is a certain Desmodur of the Bayer articles, the so-called Desmodur TT is such a dimer of a (177) di-isocyanate.

Now, that concludes the teaching as far it goes to this point.

And now comes examples.

Q. Perhaps I can shorten your explanation just a little by asking you is example 1 a polyester material? A. Example 1 is a polyether material.

Q. I beg your pardon. I was thinking of example 2. Example 1 is a polyether material but using a diol; is that correct? A. Example 1 uses a diol as the initiator and uses a mixture of ethylene oxide and propylene oxide as a chain former, and then uses TDI as the di-isocyanate in certain proportions.

Q. Then example 2 is the polyester? A. Example 2 uses a cross-linking unit, namely, a pentaerythritol and uses propylene oxide as the chain former, and then uses phthalic acid. In fact phthalic acid ester is used as a chain former, as an extender.

This is a polyester-polyether combination. The original chains are polyethers, and then they are put together via polyester bonds.

Q. All right. Then what is example 3, doctor? A. Example 3 uses, again, propylene oxide as the (178) chainformer and uses pentaerythriotol as the cross-linking point, that is a tetrafunctional tie point-former, and uses

TDI as di-isocyanate. In other words, this is a polyether di-isocyanate composition.

Example 4 goes back to a diol. In fact, it goes to the same diol—sorry. Not quite to the same—to a diol similar to the one in example 1, and reacts it with MDI, the other di-isocyanate which is usually employed.

Then this product is further condensed with TDI. In this case another trifunctional product was added, namely, triethylamine, and this is a catalyst.

Q. You refer to the triethylamine as a catalyst? A. Well, probably it is a catalyst.

And then this polymer obtained in this manner, that is, obtained from the diol, with MDI and TDI, was compounded like a rubber would be compounded with the carbon black and other processing ingredients and was cured, and the properties were determined and compared with the properties, the mechanical properties of the rubber were determined and compared with the properties of a standard rubber of the market, and that is example 3.

Q. All right. A. Then in column 5-

Q. I think you meant example 4, didn't you? (179) A. Then—Excuse me. In fact, this was example 4, or this was example 4, a part of the teaching of example 4 because on the next page there are additional tests reported, whereas the tests on page—sorry—whereas the tests in Column 4, at the bottom of Column 4, refer to the mechanical properties, the tests on the top of Column 5 referring to other properties, swelling characteristics, behavior at low and at high temperatures.

In all cases comparison is made with a standard commercial rubber known as paracril 18-80.

Now, comes example 5 starting on Column 5, line 24, and this example teaches the use again of a copolymer of ethylene oxide and propylene oxide initiated by a diol or a glycol, so that we have a long chain in which ethylene and propylene units are alternating, and the chain

has a molecular weight of 2,000 and has hydroxyl groups. There is one hydroxyl group in each hand.

This material which is a liquid, at elevated temperatures, is mixed with TDI, with one of our di-functional isocyanates, and the resultant product was reacted to give a superior elastomeric polymer similar to those of the preceding examples.

There is no teaching as to the exact character of this material, and apparently example 4 was selected to (180) make a series of measurements which would permit to compare the rubbers of this invention with standard rubbers of the market.

Now, the next short paragraph really only indicates that the high molecular weight intermediates are polyethers, which was obvious, and there are terminal hydroxyl groups.

Of course, they have terminal hydroxyl groups. If one starts the reaction with a glycol or with a trifunctional material, that is.

So it says that these polymers are also believed to contain terminal hydroxyl groups.

Then comes a teaching about the ratios. The polyglycols and di-isocyanates are combined on approximately an equimolar basis.

- Q. What does that mean to you, Dr. Mark? A. On an equimolecular or equimolar basis means that the number of NCO groups and the number of OH groups which are introduced in the reactor is about the same size.
- Q. Would you expect a product to terminate in hydroxyl groups or NCO groups? A. Well, at the end, on the average, statistically, if they are exactly equimolar, half of the ends would be hydroxyl groups and half of the ends will be NCO groups, (181) and then, of course, it is said, when the elastomer is too soft, additional di-isocyanate is added in order to get more cross-linking.
 - Q. Would this be the excess? A. That's correct; yes.

And when it is too hard and resinous; in other words, when, instead of a rubber we get a resin, which is not the purpose of this invention, then less of the di-isocyanate should be used.

And then, at the end, he says that polyglycol should preferably be heated prior to the reaction, at least until it is in liquid form.

Many of these materials, like the polyethylene glycols, or polypropylene glycols actually are solid, flakey, white flakey solids.

Of course, in order to get them to react they have to be melted.

Q. That is common, is it? A. Yes, sure. That's obvious.

And I think this is the teaching of the patent, essentially, as far as I can see it.

- Q. We have been talking here, or focusing, if I may, on the propylene oxide triols and tetrols in so far as the Heiss patent is concerned, and their reaction with disocyanates? (182) A. That is correct.
- Q. I would like you to look back at the examples in Dr. Price's patent. Example 1 is a glycol so that it is not a triol or a tetrol; am I correct? A. Example 1 is a glycol which contains ethylene oxide and propylene oxide; yes.

Q. But it would be a diol? A. That is correct.

Q. And example 2, of course, is a polyester which we are not really concerned with in so far as what the Heiss patent covers, but example 3 is the reaction of propylene oxide with pentaerythritol, which is a tetrol? A. Yes, sir.

Q. And the isocyanate is TDI? A. That is correct.

- Q. In other words, this is within the Heiss definition of its invention, am I correct? A. Well, I would use a different terminology.
- Q. All right. A. I would say that this is a case, or this is an experiment which shows how to react, how to build

up a polyether polyol with the aid of pentaerythritol and propylene oxide and then react this polyol with a diisocyanate.

Q. Well, yesterday you talked about Claim 3 in the (183) Heiss patent, if you recall? A. Yes.

Q. You were comparing it to the Windemuth reference? A. That is correct.

Q. What I meant was that Claim 3 of the Heiss patent calls for propylene oxide triol or tetrol, wouldn't it? A. That is correct; yes.

Q. Reacted with isocyanate? A. Yes.

Q. That was what I meant. A. Yes. That is correct.

Q. And then example 4. Again we go back to the reaction of a diol, don't we? A. Now, in example 1, the material disclosed is ucon fluid 75-H-1400, and this is a diol.

In example 4 what is specified is ucon 74-H-1400.

I don't know whether they are identical or what the difference is. As I sit here I don't know.

Q. Do you have any opinion? A. My opinion is that it is also a diol but I may be wrong, but to the best of my knowledge right now it is another diol.

Q. And then example 5 is the reaction again of a gly-

col which is a diol, isn't it? (184) A. Yes.

Q. What I am getting at, doctor, is that example 3 of the Price patent is the only one that shows the reaction of a propylene oxide tetrol? A. That is correct.

Q. What was the reaction of example 3 run in? A. In

alpha-chloronaphthalene.

Q. And what is alpha-chloronaphthalene? A. It is a

liquid, an organic liquid.

Q. Is it, in this case, used as a solvent? A. Well, I would guess that since it has a high boiling point—I don't know right now what boiling point it has but I am sure it has.

Q. I can tell you it is 263 degrees centigrade. A. I would have guessed that it was above 200, and since the temperature is not specified here exactly, in any event, I am sure that the alpha-chloronapthalene was contained in the final product.

Q. Then maybe you can compare alpha-chloronaphthalene and the aroclor that we have been talking about in so far as the reaction of example 3 is concerned? A. Yes. Alpha-chloronaphthalene is a high boiling inert liquid just like aroclor is one or another of the inert high boiling liquids which are disclosed in the Heiss patent. (185) It is of the same type. So it is a plasticizer.

Q. Is is also a plasticizer? A. Sure.

Q. And therefore would you agree with me that the alpha-chloronaphthalene in example 3 of the Price patent performs the same function as the aroclors in the Heiss patent? A. I would say that it performs a similar function. I don't want to use the "the same" because quantitatively it may not be the same, but it performs a similar function.

Q. They are very similar aren't they? A. (No response.)

Q. I am not sure you heard me, doctor. I said they are very similar, the function of the alpha-chloronaphthalene and the aroclor are very similar in so far as the reaction of example 3 in Price are concerned. A. I said already, in order to repeat my position, there will be differences in degree. Otherwise the function of the alpha-chloronaphthalene in example 3 is similar, and if you wish, very similar to the function of the various plasticizing non-reactive organic liquids in the various examples of Heiss.

Q. Thank you.

Now, have you looked at the claims in Dr. Price's (186) patent? I don't think that I want to ask you

to go through them all as you have the specifications; I just wonder if you are generally familiar with them. A. I have looked at them but of course—

- Q. Well, the question I would really like to ask you is whether or not the alpha-chloronaphthalene appears in any of the claims of the Price patent. A. I really would have to read them in detail. I haven't read them with enough attention to be able to answer this question.
- Q. Well, I don't want to take the court's time while you read. Perhaps you can consider it over the lunch hour.

(187) By Mr. Sweeney (Cont'd):

- Q. You testified yesterday, and we reviewed it again awhile ago, and you said that there were three components in the Heiss invention, and one of them you mentioned as being the aroclor? A. It is not always aroclor.
- Q. Well, all right. The organic solvent? A. The plasticizer.
 - Q. The plasticizer? A. Sure.
- Q. Do I get from your testimony that you feel that the plasticizer is essentially or essential to the Heiss patent? A. Yes.
- Q. Then I would like you to look at Column 3 starting at line 69 where it says: "In either case the products are relatively brittle resins which may be plasticized and rendered more flexible by effecting their preparation in the presence of a non-reactive organic material . . ." A. Yes.
- Q. Does that say that the non-reactive organic material is required? (188) A. Well, I think it says here that the brittle resins, if the brittle—

You see, the reason why I am—is that which may be plasticized. If they are too brittle, you plasticize them.

Q. What I meant was it doesn't say that they have to be plasticized. A. Well, they have to be plasticized

when they should be useful.

Q. You are reading something into the specification apparently. I just want you to tell me whether or not it says that they may be used, that they don't have to be used. A. Well, I think I can best make my position—or I have been trying to make my position more understandable by the following:

If we go through the materials which are needed in order to practice the Heiss invention, then in Column 2, 44, we find that illustrative examples of suitable isocyanates, and so on and so on, which may be employed—that is his language, his teaching in respect to the isocyanate component.

Q. Did that mean that they had to be employed? A. I am just, for the time being, not interpreting what

it means. It is his language.

Then we go to the top of 4, and there he uses (189) almost the same words: ". . . as illustrative examples of non-reactive organic liquids and solids which may be used . ." So what is the difference between "may he employed" and "may be used"? Nothing.

Now, it is certainly clear that we cannot leave out the isocyanates if we want to practice the Heiss invention.

Now, since Heiss uses the same teaching, verbally the same teaching, for the introduction of the di-isocyanate as he does for the introduction of the non-reactive organic liquid, which is the plasticizer, I conclude that the importance and the necessity of those two ingredients are of the same order. That is the reason really which compelled me to testify the way I did.

Q. All right. Then would you take a look at Column 9, line 75, the top column, 10, down to line 7? A. Yes. And when I read that, what it tells me is don't omit

them. It is undesirable to omit them.

Q. It doesn't say don't omit them, though, does it? A. Well, it says, "It is desirable to have them there."

Q. Which means that they might not be there? A. Well, again I must say, I am reinforced in my interpretation by comparing the way in which Heiss tells me what kind of isocyanates I should use and what kind of non-desirable, (190) non-reactive organic liquids I should use.

He uses the identical words. And this is the reason why I said that this is another component.

Q. Then will you look at Column 4, line 20 in that sentence which goes down to about line 24? A. (Witness complies.)

Q. There it says that the adducts may be preformed, doesn't it, and then added to the non-reactive organic liquid? A. Yes, but the adducts are the condensates of the diol or triol or tetrol, or whatever it is, and the prepylene oxide.

Q. But you have used the word "adduct" yourself for the final reaction product? A. Yes, but if he would here use—

You see, in this-

Now it says, it starts by saying "The above-described adducts may be preformed and then added to the non-reactive organic liquid or solid and converted to a more highly polymerized state" by probably heating, or something else, "or they may be formed in situ and then converted to a higher state of polymerization."

Now, that shows that the adducts must be the condensates of whatever he uses, the diol or tetrol or ethylene oxide.

(191) Q. Well, the reaction between the propylene oxide triols or tetrols and the isocyanates is a polymerization, isn't it? A. Yes.

(192) By Mr. Sweeney:

Q. Dr. Mark, before lunch I had asked you to look at the Price claims and to tell us whether or not any of the Price claims include the alpha-chloronaphthalene that is used in example 3. A. None of the Price claims includes any plasticizer such as the alpha-chloronaphthalene in claim 3. In fact, the entire patent does not use anywhere the word "plasticizer" nor is there any allusion to desirability of a plasticizing action.

Q. Is the use of a plasticizer, such as the ones we have been talking about in polymer chemistry, a common

thing? A. Yes.

Q. Would you please look again at the Heiss patent. I ask you in particular to look at the second full paragraph in column one, which begins at line 13, I guess. In your discussion yesterday you didn't allude to that paragraph and I wondered if you would discuss the materials that are listed there for us. A. In this paragraph are listed the following applications of the products of the invention.

(193) For casting or pouring into intricate forms or crevices. That is one application.

Then there is adhesives, which is another one. As protective coatings is one.

Components of insulating varnishes is one.

Impregnants for fibrous material to make water resistance, flame resistance and improved hand.

Insulating and electric elements and transformers, capacitors, electric terminals or bushings, cables or other electric devices and as foamed-in-place resins.

Q. Thank you. Are those products which are commercially manufactured? A. Yes.

Q. Are urethane materials used in manufacturing those products? A. Some of them, yes.

Q. Would you categorize them as useful products?

- Q. Yesterday you discussed the Windemuth patent, which I believe is plaintiff's exhibit 5, and in doing so I believe it was your testimony that the propylene oxides and I think you mentioned butylene oxide and I think you said one or two other oxides were disclosed because the patents said alkylene oxides. (194) A. That's correct.
- Q. I am not trying to put words in your mouth; I am just trying to shorten this up. A. That is correct.
- Q. Does Windemuth say propylene oxide anywhere in the patent? A. Windemuth does not specifically use propylene oxide by naming it, but he does disclose the use of propylene oxide derivative, namely, of epichlorohydrin and in describing this use he refers to a terminal alkylene oxide group. That is in column 4, lines 13 and 14.

Because epichlorohydrin is a propylene derivative it terminates in an alkylene oxide group.

Q. What I asked you—and I think you answered it and gave me additional information—was whether or not Windemuth at all in the patent specifically says propylene oxide anywhere within its four corners. A. It doesn't use the word propylene oxide.

Q. This morning before lunch we talked about, what shall we call it, the hydrolylic quality of the ethylene oxide urethanes and the propylene oxide urethanes. Would you compare those two insofar as their affinity for water is concerned? A. Under otherwise equal or similar conditions, polypropylene oxide materials are less swelling, less (195) moisture uptaking than the corresponding polyethylene oxide materials.

Q. For the purposes of, shall we say, commercial urethane foams and elastomers today, is it desirable today to have a less water-loving system? A. For foam specifically I think it is in most cases desirable.

For, for instance, the treatment of fabrics, it is not desirable. In fact, it is undesirable.

Q. But there are instances in commercial urethane use where the more or less hydrophobic materials made from

propylene oxide are more desirable? A. Yes.

Q. What does Dr. Windemuth say about the products that he gets from reacting his polyglycol ethers insofar as their water loving qualities are concerned? I think I might be able to help you by calling your attention— A. Can I just enumerate a few instances? Not all of them.

Q. All right; go ahead. A. In example one he says in column 5, line 10 or 9 and 10, a light yellow resin is obtained on cooling which is insoluble in hot and cold water.

In example two and three and four he again (196) characterized his products as being insoluble in water.

In example 5 he discusses the properties of eight products in a table on the top of column 6, some of them are soluble, others are insoluble in water.

Example 6 produces a product which is insoluble in water, but swells in water. It has a swelling capacity as

spelled out in line 44 of this column.

I think that shows that he can just—depending upon the conditions under which the material is made, the proportions of the ingredients and so on—he can vary the reaction or the behavior of his product against water.

- Q. Is there a difference between a urethane material that is soluble in water and one that swells in water? A. Sure.
- Q. Maybe you would describe that for me. A. Well, I mean cotton swells in water. My handkerchief swells in water; but is not soluble.
- Q. Would a swelling quality, for example in a urethane foam, be a problem in water swelling quality?

A. Well, I think it depends on the use of the foam. If one wants to use a polyurethane foam, let us say, to make sponges with which one can wash windows and cars, a certain degree of water swellability will be actually desirable.

Of course, on the other hand if one uses the foam (197) in the upholstery field, then probably one would prefer a material which does not swell in water.

- Q. Do you know whether the propylene oxide based foam swells in water? A. Less. It swells less.
- Q. Will it swell at all? A. Well, of course everything swells a little bit. I don't know, maybe the moisture absorbtivity is very low, but any way I can make a statement in general that propylene based foams will swell less.
- Q. In the context of the commercial use for upholstery, propylene based polyurethane foam doesn't swell enough to cause a problem? A. Again I would like to say that they swell less, substantially less.
- Q. What does Windemuth mean in column two in the paragraph which begins at line 18 where he talks about these reactions of polyglycol ethers and he gets down to the bottom in the next to last sentence and he says the plastics are of interest because of their swelling properties? A. Well, I presume in this specific case he thinks about such uses as impregnation of fabrics and certain coatings, ceatings on wood, for instance, they must swell a little bit in water, otherwise in the long application (198) they will peel off.
- Q. He is talking about the polyglycol ether reaction with iscovanates? A. Yes.
- Q. And he says that these plastics swell, which is what we have been talking about. A. Yes.
- Q. I presume, therefore, that he is teaching that these plastics will swell in connection with water? A. Oh, sure. Usually if there is no other word used than

swelling, usually it refers to swelling in water, although, of course, there are certain swellings, for instance in gasoline. Where you refer to, the plastics are of interest because of their swelling properties, it really doesn't say in what they swell.

Q. It does— A. But then it says, it continues, now, for instance, it is possible, according to the invention, I

am now reading, your Honor-

The Court: I am following.

A. Line 33. To prepare rubber elastic products which are completely indifferent in aliphatic hydrocarbons however swell in contact with water.

For instance, one wants rubbery materials not to swell in hydrocarbons if they are used as sealants in a system where (199) they come in contact with gasoline and these materials could be used for this purpose.

Mr. Sweeney: Thank you, Doctor.

Q. Yesterday you were asked to give your opinion as to a quote which Mr. Kelton read to you and now I am not trying to reread the quote, but it was to this effect, Windemuth discloses only ethylene oxide and I helieve you said that that was incorrect. A. Yes.

Q. My question is related to the one I asked you awhile ago. It is true that there is no specific disclosure of propylene oxide in Windemuth, isn't it? A. Again I must say he doesn't use the word propylene oxide. He uses alkylene oxide which for every chemist comprises propylene oxide and more than that he describes the reaction of his products with a propylene oxide derivative.

Q. All right; we have been through this before and I guess there is no use in burdening the record. A. Yes, sir.

(201) By Mr. Sweeney (cont'd):

Q. Now, if you would, Doctor, turn to the Sokol reference, which I believe is plaintiff's exhibit 6. Does Sokol have anything to do with urethanes? A. No.

Q. If you would then, please, look at the English translation of Dr. Bayer's article, which is plaintiff's exhibit 7-B. The chart, Doctor, which is marked as plaintiff's exhibit B. A. That is this chart here? (indicating)

Mr. Sweeney: Yes.

Q. With reference to plaintiff's exhibit B, which shows the network you described this morning, is there any suggestion in that chart that the units are polyethers? A. No.

Q. Are there any trifunctional compounds disclosed in the Bayer article? A. Yes.

Q. What are they? A. If I may draw your attention to page 41. We go (202) down to the first section which begins on that page.

Q. Is that the one that begins with "this reaction"? A. That is correct. "This reaction can be utilized for production of new and highly stable foam substances. By mixing a polyester from a dycarboxylic acid and trivalent alcohol." That is one place where a trivalent compound is disclosed.

Q. That disclosure relates to polyester, of course? A. Sure.

Q. There is no disclosure there of propylene oxide. A. No, no, no, This is the principle, this is a matter of disclosing a principle. Whether this principle is finally embodied by one or another material is not here discussed.

Now, then, may I draw your attention to page 50 and on page 50 the last section in the fourth line—

Q. Is that the one beginning "the combination"? A. That is correct. Castor oil is used and we know that castor oil is a trifunctional—is a triol.

Q. But it is also a polyester, isn't it? A. Yes, sure. But in my answer to your questions as to whether in the Bayer article trifunctional polyols are mentioned is yes, they are mentioned.

Q. I wasn't arguing with you. (203) A. No, no, I just wanted to—I didn't want to argue with you either.

Q. I just meant that castor oil is a polyester. A. That is correct, yes.

Q. I am not sure, but I think this morning you said that there were two polyethers shown in the Bayer article; am I correct? A. Yes.

Q. Just to clarify my mind would you point them out to me? A. Well, I think what I said—at least what I hoped to say—is the following: in table 2 the eighth and ninth ingredient—

The Court: What page is that, Doctor? The Witness: That is on page 16, your Honor.

A. The eighth and ninth ingredient, which have melting points of 120 and 124, are monoetherdiels. They have one ether bond and two hydroxyl groups so they are not a poly, they are a monoether diel.

But farther down a compound which has a melting point between 208 and 212 is a diether diol or in our nomenclature a polydiol and those are the two materials which they have prepared and characterized and used.

Q. Which you characterize a diol and a polydiol; (204) am I correct? A. Yes, I think we always have done that here.

(205) Q. Those are the only polyethers disclosed then? A. Yes.

Q. There is no disclosure of propylene oxide based triol or tetrol, is there? A. No.

Dr. Herman F. Mark, for Plaintiff, Re-direct

- Q. Dr. Bayer was a co-inventor with Windemuth in the Windemuth patent, wasn't he? A. That's correct, yes.
- Q. If I may ask you to turn to the Hill patent, which is Plaintiff's Exhibit 9, the reactions in Hill are all with glycols, aren't they? A. Yes.
- Q. Are you aware that the Hill patent was cited against the Price patent as prior art? A. I think I remember, yes.
- Q. Do you find any suggestion in Hill at all about using triols or tetrols? A. No.
- (207) Q. What I meant was is there a claim in the Heiss patent which would cover a castor oil isocyanate reaction product. A. No.

Re-direct Examination by Mr. Kelton:

(209) Q. Mr. Sweeney was implying, as I understood him, a comparison between Price and Heiss, the patents. I wonder if you have any comment you could make on the comparison of Price and Heiss, so far as plasticizers, their significance or in that general area. A. Well, there is a significant difference. When a chemist reads the Heiss patent, he becomes aware of the fact that there exists a considerable degree of preoccupation of the inventor with a plasticizing ingredient. It is introduced on the top of column four with the same weight and, in (210) fact, with the same words as is introduced the diisocyanate in column 2, line 44.

Again, two other places later on, in columns 10 and 11, attention again is drawn by the inventor on the importance and desirability of the plasticizer.

All examples contain it. Several examples—in fact, examples 11, 12, 13, 14 and 15 all are completely devoted to the study of the amount and the character of the plasticizer.

In other words, the plasticizer or the nonreactive organic liquid figures as a major part in the Heiss patent.

In the Price patent, it is not even mentioned. The only ingredient which has plasticizer characteristics is the alphachloronaphthalene in Example 3. Nowhere else is there any word or any mention of a plasticizer or of the desirability of a plasticizing action.

Mr. Kelton: I have no further questions, your Honor.

The Court: Mr. Sweeney, anything further?

Re-cross Examination by Mr. Sweeney:

Q. I think I have already asked you this but, castor oil doesn't have any propylene oxide, does it? A. No.

Q. Referring again to the Price patent, in example 3 is (211) the only example that discloses the uses of a propylene oxide pentaerthritol condensate, am I correct? A. Yes.

Q. That example uses alphschloronaphthalene as a plasticizer? A. Yes.

(220) CHARLES C. PRICE, called as a witness for the plaintiff:

(221) Direct Examination by Mr. Kelton:

Q. Would you please state your name and residence? A. My name is Charles C. Price. My residence is 118 Hilldale Road, Lansdowne, Pennsylvania.

Q. What is your present position? A. I am Benjamin Franklin Professor of Chemistry at the University of

Pennsylvania.

(222) Q. Can you fill us in a little on your education? A. I went to college at Swarthmore College, entering in 1930, and got my Bachelor's degree in Chemistry with high honors in 1934.

I then began my graduate work at Harvard University in September of 1934 and completed my PhD, my doctoral degree, in June of 1936.

Q. Dr. Price, can you outline for us your experience, your employment? A. My employment has been—

Q. Following your receiving your PhD from Harvard in 1936? A. It has been in three universities. I went in the fall of '36 to the University of Illinois, where I first served as a post-doctoral research assistant to Professor Roger Adams, and then was put on the teaching staff and, first, as an instructor, associate, assistant professor and in 1942, I became associate professor at the University of Illinois.

At the end of the war, I left the University of Illinois in January of 1946 to go to the University of Notre Dame as professor of chemistry and chairman of the chemistry department. I served as chairman there, except for a brief leave of absence of a year or so when I ran for Congress (223) in the United States Senate, returned to the chairmanship, but shortly thereafter in 1954 left Notre Dame to go to the University of Pennsylvania as Blanchard Professor of Chemistry and chairman of the chemistry department at Pennsylvania.

I did resign my administrative duties as chairman in 1965 when I was president of the American Chemical Society.

Q. Doctor, do you have any honors and awards? A. Yes. The first and perhaps one of the best is the American Chemical Society award in pure chemistry, which I received in 1946. This is given annually to the young chemist, under 36, who has published the most distinguished or exciting research. I received this when I was 33 years old in 1946.

For the research I did during the war, which involved some work on synthetic rubber, some work on chemical warfare agents and some work on drugs for malaria, I received an Army-Navy certificate of appreciation in 1947.

In 1948 I was awarded the Indiana Junior Chamber of Commerce Young Man of the Year award.

In approximately 1955, I received the Honor Scroll of the Chicago Chapter of the American Institute of Chemists.

And in 1963, the Honor Scroll of the Philadelphia Chapter of the American Institute of Chemists.

In about 1965 or 1966, the Honor Award of the Philadelphia Section of the American Chemical Society. (224) That I think covers most of them.

Q. Do you have publications, Doctor? A. Yes, sir. I have published approximately 250 or a few more than 250 scientific papers, either alone or with my students.

I have approximately 20 patents on a variety of different subjects, and I have written or co-authored four books. The first was a brief course in organic chemistry, which was an elementary college chemistry course.

The second was a book called "Reactions of the Carbon Double Bond," which was published in 1946 and dealt extensively with the chemistry of polymerization reactions.

Approximately 1955 or 1956, I published a book on sulfur bonding, which had to do with the effect of sulfur in organic compounds on their chemical and physical properties.

In about 1970, I published a book called the "Geometry of Molecules," as part of a chemistry-biology inter-phase series, and I am now involved in writing a book for a bench mark series on the origin of life.

Q. You stated in connection with your previous answer during the time you were president of the American Chemical Society—when was that? A. Well, I have been a member of the American Chemical Society ever since I finished my doctorate degree, I guess in (225) '36 or '37. I have served in many capacities for the Society and I served as president of the Society in 1965.

I have also had other offices in the Society. I instigated and was the first chairman of a committee on chemistry and public affiairs for the American Chemical Society.

I also belong to some other scientific societies, The American Association for the Advancement of Science, and I was the vice-president of that organization for chemistry in 1971.

I am also a member of the British, German and Swiss Chemical Societies.

- Q. At the time you were president of the American Chemical Society, about how many members did they have, as you recall? A. They had just passed the 100,000 mark in members.
- Q. Could you just tell us briefly what your areas of chemical interest are? A. My most fundamental basic interest in chemistry that I guess pervades almost all of my publications has been an interest in the relationship of the structure of molecules to their chemical reactivity.

Within that broad, very broad test, I have done work extensively on polymers. I guess if I had to select any one area in which I had done the most work it would be in polymer (226) chemistry, both in the vinyl polymerizations, that is the polymerization of compounds with carbon double bonds, and in the preparation of polyethers.

I would guess that that constitutes somewhere around 30% of my total research publications.

Another 30% roughly would be related to the synthesis and properties and the chemical reactions and the preparation of many other organic compounds.

About 20% of my work has been involved in what I would call fairly theoretical studies of the kinetics and mechanisms of organic reactions, and about 20% has been involved in areas related to biochemistry such as the treatment of malaria, the treatment of cancer, and some of the very important reactions of proteins and nucleic acids that are such important materials in living systems.

I might just add that the one other thing that I have done quite a bit of in the scientific line, in addition to writing my own things, has been editing. I have been one of the editors of—one of the original editors of the Journal of Polymer Science. I have been on the editorial board of Chemical Reviews, and of the Journal of Organic Chemistry.

I was on the editorial board of a series of books called "Concepts in Chemistry" published by the (227) Rand Press. I was the Chief Editor of a series of books called "The Chemistry-Biology Inter-Phase Series", published by McGraw Hill & Co.

Q. Can you tell me whether or not you are or have been a consultant? A. Yes. I have been a consultant in two different areas in industry.

I have been a general chemical consultant for the Socony Mobil Oil Company from about 1942 or '43 to about 1956.

I have been general consultant for the Hercules Powder Company from about 1942 until about 1966:

I have been general consultant for the Eli Lilly Pharmaceutical Company from about 1942 through the present.

I have been a general consultant to the General Tire and Rubber Company from about 1946 through the present.

In addition to my industrial consulting, I have also been involved in considerable activities of this kind with the government. I was on—for the National Research Council, I served on their advisory committee to the Quartermaster Corps for many years, and I served on and in fact for many years chaired the ACS, American Chemical Society Advisory Committee to the Chemical Corps.

I have also been on the Study Sections, which is (228) an advisory group, to the National Institute of Health in relation to cancer and I served as the second chairman of the National Science Foundation's Divisional Committee on Mathematical, Physical and Engineering Sciences.

I think that covers most of my major consulting work.

Q. Dr. Price, I show you Plaintiff's Exhibit 1, which is a patent to Herbert L. Heiss, and I ask you to look at Claim 3 of that patent and I will ask you whether you or anyone working under your supervision ever prepared a condensate of propylene oxide with an alcohol having three to four OH groups, and then reacted that condensate with an excess of polyisocyanate?

Mr. Sweeney: Your Honor, Dr. Price is a fact witness, I think, in the main.

Mr. Kelton: I am asking him.

Mr. Sweeney: I would object to that question as leading. I said the other day that with an expert I can see where it is necessary, but with a fact witness, if the Court please, I think the rules of evidence should apply. That is a leading question.

The Court: All right, Mr. Kelton.

Mr. Kelton: I will withdraw the question, your Honor.

(229) Q. Dr. Price, have you made, as you recall, any invention in the field of polyether urethane rubber? A. Yes, sir.

Q. Do you remember about when? A. Yes. The idea on which that invention was based was at the end of January 1949, and we made the first rubber balls out of polyurethane polyether by early April 1949.

Q. Doctor, can you tell me a little bit more about the idea in January 1949 to which you referred on which you indicate the invention was based? A. The basic idea involved the concept that polypropylene oxide chains would make an excellent—if incorporated into a network structure—would make an excellent rubber material.

When I first got this idea at the end of January in 1949 that this would be a desirable rubber, the next problem was how one could make such a network structure.

Q. Before you get to that point. Doctor, let me ask you: You said the end of January 1949. How are you sure of the date? A. As a part of my duties as a member of the National Research Council Committee advisory to the Quartermaster Corps, I was asked to organize a research conference to discuss a problem which was a very serious problem, with the (230) military at that time of how to make rubber which would be useful at very low temperatures, at very high altitudes. We were beginning to get bombers that flew very high. We were

considering the problems of the defense of this country from Arctic attack, so there were several areas in which the problem of developing rubbers which would serve wide useful purposes at very low temperatures had become an important national objective.

The state of the art was not well organized at that time and we thought it would be useful to try to bring together a group of the most competent experts we could find in this country to exchange their information and ideas about the various ways one could try to solve this problem.

As I say, I was asked to chair that, and the conference was organized and we brought together a group of roughly 12 or 15 scientists to meet about three or four days in Washington at the—I have forgotten the name of the hotel—one of the hotels in Washington.

The meeting was, in fact, very successful in that the wide variety of physicists and chemists, theoreticians and practical people met there in this small group, did stimulate a lot of ideas.

One of the ideas that occurred to me as a result of this was, in fact, this very idea that the characteristics (231) simple propylene oxide, polypropylene oxide molecules suggested to me that if they could be incorporated into a network structure, they would, in fact, be a very useful rubber.

It was this idea that I mentioned and discussed at the conference and took home to Notre Dame to look in the literature to see if there was in fact any way to accomplish this.

- Q. When was this conference held? A. It was about the last three days of January 1949, the 28th, 29th and 30th, T believe.
- Q. You said that you went back to Notre Dame with this idea, as I recall. What did you next do, as you recall? A. I was, of course, well familiar with the

classical way of making a rubber network structure, which was to take the long polymer chains that are present in natural rubber or in the various diene synthetic rubbers and then cross link those by vulcanization into a network structure.

I knew at that time nothing, or virtually nothing, about polyethers, but a look in the literature revealed that no one had ever been able to make a high molecular weight long chain polyether of propylene oxide. The highest molecular weights I could find recorded anywhere in the literature was 3,000 molecular weight, whereas, to make (232) a good rubber by the classical vulcanization procedures, one needed a molecular weight of at least 25,000.

So, it became immediately obvious that the conventional method of trying to make such a network was not going to be applicable on the basis of the present state of the art.

However, there had been disclosures of the Vulcollan rubbers by Otto Bayer, in fact, they were already beginning to be introduced into commerce at that time employing the disocyanate technology to make urethanes out of low molecular weights, diols and polyols.

So, the alternate idea occurred to me then that perhaps the way to try to solve this problem was to apply the Vulcollan type idea to polypropylene oxide rather than the conventional sulfur vulcanization technique.

That is what we proceeded to do, and by "we", I mean myself and my student Ralph Herbst, who it happened was just finishing his doctoral dissertation in Norte Dame in March of 1949. He already had a teaching job, which was to call him to Utica, New York, in September, and so it occurred to both of us that it would be interesting if he could carry out the experiments which arose in my mind out of this idea.

- Q. Did you carry out work or have it done for you in (233) this connection? A. Yes. I did arrange for General Tire to provide a stipend for Ralph Herbst to do this work.
- Q. It was Dr. Herbst that did the work? A. That is correct. He did the experimental work in the laboratory.
- Q. About when in the year 1949 did you first take this up with Dr. Herbst? A. It would have had to have been some time in March of 1949.
- Q. Do you recall what you did? Well, let me put it this way, Doctor.

You told me you got a stipend from General Tire for this work. Did you get the stipend before you started Dr. Herbst on the work? A. Well, actually, Herbst started in March. After I had discussed this idea orally with Mr. Gilbert Swart, research director of General Tire, I did not have a formal written request and authorization from Mr. Swart until April of 1949. (234) Q. Can you tell me how and when you proceeded with Ralph Herbst in connection with this project? A. Well, I called him in to ask him if he would be interested in carrying out these experiments. Protheses

A. Well, I called him in to ask him if he would be interested in carrying out these experiments. By these experiments I meant the basic idea which I outlined to him was to make polypropylene oxide with 3 or 4 more hydroxyl groups to serve as the crosslinks, for the network; to react such glycols, which we would have to make ourselves. These trihydric and polyhydric polypropylene oxides were not available at that time.

They did later become articles of commerce, but in 1949 we had to make our own triol and tetrol alcohols, polypropylene oxides. So the first step was to make the branched polypropylene oxides and we selected pentaerythritol as the main branching component and we selected sodium as the catalyst to condense propylene oxide with pentaerythiritol.

I then outlined for him two possible-

Q. As I understand it, you were attempting to make a condensate of propylene oxide and pentaerythritol? A. Exactly so.

- Q. Okay. A. The second step in our procedure would then be to convert this low molecular weight liquid into a rubber network by the use of the hydroxyl groups and we first elected (235) and I outlined two ways to do this; one was the use of the di-isocyanate technique to link the hydroxyl groups through urethanes and the other was to use a dibasic acid to link the hydroxyl groups of this polypropylene oxide by ester links. So we outlined two different strategies for converting the branched chain polyol into a network structure. One through polyurethane and one through polyester and he, in fact, did both of those before he left in September.
- Q. When you refer to a dibasic acid, is that the same as a dicarboxylic acid? A. Exactly.
- Q. In other words, it has two carboxylic acid groups in it? A. That is correct. It is another name for a dicarboxylic acid.
- Q. Dr. Price, I show you a letter—a copy of a letter on the stationery of University of Notre Dame, dated April 7, 1949, and which bears Price's note on the upper lefthand corner "Price's exhibit 7" and bears also a stamped number 10.

Can you tell me what this document is? A. This is the letter that I wrote to Mr. G. H. Swart, the director of research of General Tire and Rubber Company, to confirm my discussions with him earlier, verbal discussions, (236) about having Ralph Herbst carry out this project of making polyether—polypropylene oxide rubber.

Q. When you wrote this letter of April 7, 1949, did it contain the writing "Price's exhibit 7" on it? A. No, sir.

Q. Did it contain the stamp number 10? A. No, sir.

Mr. Kelton: I offer in evidence as plaintiff's exhibit 16 a copy, photocopy of the letter of April 7, 1949 which the witness has just identified as a letter written by him.

Mr. Sweeney: No objection, your Honor.

The Court: It will be received.

(Plaintiff's exhibit 16 was received in evidence.) Mr. Kelton: I would point out, your Honor, that through the course of the years these documents have been used for many purposes and the handwritten "Price's exhibit 7" was added and the stamped number represents the production number in this case when we produced it.

The Court: All right.

- Q. Plaintiff's 16, as I understand it, Dr. Price, and correct me if I am wrong, is a request for the appropriation or provision by General Tire of another \$1500 to cover Dr. Herbst's work. (237) A. That is correct. I did already have funds from General Tire at Notre Dame, which were being used to support other students in their work on vinyl polymers, so when this project came along, I had no money to support Herbst so I had to get extra money and that is, therefore, the reason why "I am therefore writing to ask whether you can appropriate another \$1500," the "another" represents the fact that I did already have money for other work and this was an additional grant to cover this particular project.
- Q. While we are on the matter of plaintiff's 16, Dr. Price, I think you have stated—I'm not sure whether you have stated who Mr. G. H. Swart is. A. I think I have, but I can certainly repeat it. Mr. Gilbert H. Swart was the research director for the General Tire and Rubber Company in Akron, Ohio. He was my major

contact for all of my activities with General Tire from 1946, when I began, until his death approximately 10 years ago.

Q. Did you have any response from Mr. Swart to your requests for another \$1500 which you made in the letter which is plaintiff's exhibit 16? A. Yes, sir; my appeal was successful.

Q. I show you a photocopy of a letter addressed to Dr. Charles C. Price, head, department of chemistry of (238) Notre Dame, dated April 18, 1949, and ask you what that is. A. This is a letter from Mr. G. H. Swart, director of research, to me as the head, department of chemistry. Notre Dame, thanking me for my recent letter regarding propylene oxide rubber.

He was very pleased of my confidence in its practical application. He talked over the financing with his management and they have agreed to appropriate a maximum of \$1500 for this special work.

Q. Is this a copy of the letter which you received shortly after the date it bears. A. Yes.

Mr. Kelton: I offer in evidence as plaintiff's exhibit 17 the photocopy of the copy of the letter of April 18, 1949, which the witness has just identified.

Mr. Sweeney: No objection, your Honor. (Plaintiff's exhibit 17 was received in evidence.)

Q. During the period of these letters, plaintiff's exhibit 16 was April 7 and April 18, was Dr. Herbst doing any work? A. Yes, Indeed. He, as I had said earlier, he finished his doctoral dissertation, he completed all that work in March and he began the investigation of polypropylene oxide rubber sometime in March.

(239) Q. About how long did he continue in this work? A. Well, he continued until very early September, right around Labor Day. His new appointment was an instructor in Chemistry at Utica College required him to

appear there for work early in September, so he left South Bend either right at the end of August or the very first few days in September.

- Q. Dr. Price, in Mr. Herbst's work in the period March 1949 through August 1949 or early September 1949, whichever it happened to be, can you tell me whether or not he recorded the work he did in a notebook? A. Yes. I remember that I had specifically suggested to him that he get a new notebook to record this particular set of experiments rather than using the same set of notebooks that he had used for his thesis, so he did keep a notebook record of the work that he did in a separate notebook from the notebooks that he had used for his thesis.
- Q. Now, you have referred to his thesis several times. Did that have any chemical relation with what you set him to do in March of 1949 and through, say, the end of August? A. No, it was a totally different search problem that he did for his thesis. He studied the kinetics of the decomposition of benzoyl peroxide, so that was an entirely (240) different subject than the one he did this special project on.
- Q. Now, you said that you suggested to Dr. Herbst that he get a new notebook for this project that he embarked on in March. Do you know whether he made entries in the notebook during the course of this work as he did it? A. Yes, I saw the notebook from time to time.
- Q. Did you from time to time have occasion to observe his work being carried on? A. Yes.
- Q. Do you have this notebook or know the whereabouts of the notebook that Dr. Herbst used for the period March to early September, 1949? A. We certainly do not know, unfortunately.
- Q. Have you ever been asked to look for it? A. Yes, indeed. It was a very crucial matter at one time

to find that notebook and we made every conceivable effort to see if we could locate the notebook.

Q. When was the first time that you looked for the notebook, if you remember? A. The first time that I can clearly recall is during the interference proceedings with the Heiss patent, which was after I had left Notre Dame; it must have been approximately 1958 or 9. We looked through my records. I (241) did not make a habit of keeping all the research notebooks at that time. It was not customary in a university for this.

In fact, for example, I have, I still have my research book from my Harvard thesis, but I do have some of them and they are neatly arranged in a drawer all in alphabetical order, but I checked again just a few days ago and Herbst's is certainly not amongst those notebooks.

T then also thought of the possibility that Herbst may not have turned it in to me in his haste to leave Notre Dame because he was very rushed at the end. I wrote to him in 1958 or 9. He had already undergone several moves. He looked through everything in his possession and was unable to locate any of his notebooks.

I then wrote back to the head of the chemistry department at Notre Dame, Dr. Andrew Boyle, who had served under me when I was there, and he made an extensive look through the files and the safe to see if by any chance it had been left there. So we made a fairly thorough search of every place that we could think of and so far as we know, we have been totally unsuccessful in locating the notebook.

Q. Were any reports other than notebook recordings made of the work that Dr. Herbst did? A. Yes, sir. Because I thought this was a very (242) exciting project, I had Herbst prepare some brief summaries of the

things in his notebooks for me from time to time during that several months period he worked on it so that I could have a report and so that I could report to General Tire on the essence of what he was up to.

Q. I show you a photocopy of a paper, dated April 19, 1949, and ask you what it is. A. This is a document, dated April 19, 1949, which in fact represents the first of those brief summary reports that Ralph Herbst handed in to me on this project.

Mr. Kelton: I offer in evidence as plaintiff's exhibit 18 the copy of the document dated April 19, 1949.

Mr. Sweeney: May I just see the document you propose to use? I am confused.

Are you calling this the report of April 16?

Mr. Kelton: April 19. The Court: April 19.

Mr. Kelton: It is typed April 19.

Mr. Sweeney: No objection. The Court: All right.

(Plaintiff's exhibit 18 was received in evidence.)

- Q. Do I understand correctly that prior to the date of April 19, 1949, Dr. Herbst had done work in this connection? (243) A. Yes. He had done a number of experiments and he briefly reports on them in this document.
- Q. Do you have any recollection of seeing any of the products that Dr. Herbst produced? A. Yes, indeed. There was considerable skepticism at Notre Dame about the possibilities of this accomplishment of converting a liquid into a rubber and so Herbst was quite excited when he made the first samples reported there which had rubbery properties and he brought some samples up and we bounced them around on my desk, and he showed them to some of the other people in the laboratory.

(244) Q. When was this in relation to the so-called progress report of April 19, 1949, Plaintiff's Exhibit 18? A. It was a few days before the report was typed and given to me.

Q. Now, you say you bounced these pieces around on your desk. With regard to those, what do you mean by you "bounced them around on your desk"? Tell me how big they were? A. The samples he made at that time were relatively small. Oh, perhaps the size of a marble. They were made in the bottom of a small test tube and he then broke the test tube away to obtain these samples.

They were small and they were not perfectly round, but they would bounce and it was quite exciting trying to decide where they were going to go when they bounced because they weren't perfectly round, but they certainly did indicate to me at that very early stage that we had, in fact, been able to convert a liquid polypropylene oxide into a polypropylene oxide rubber.

Now, there were other samples which he made reported in there which were not rubbery, which were hard, and for what I was looking for they weren't as exciting. They were hard and brittle samples, but I particularly remember the rubber sample because it was what, in fact, we were looking for.

(245) Q. Did you show these balls or any of them, these pieces, to anybody at General Tire as you recall? A. Later on. I don't know how much later. Probably a few weeks to a month later Herbst did at my direction make a considerably larger sample of this same material that formed the rubbery ball, perhaps a ball the size—maybe the size of a tennis ball, a hundred grams approximately, and we did—I did take that sample to General Tire to try to show them that we had made a rubber out of this project.

Q. Now, sir, with regard to Plaintiff's Exhibit 18, I notice that there is a heading in handwriting "Progress Report" and then "To: Dr. C. C. Price", which is crossed out, and there is handwriting added or below. Can you tell me the occasion for that? A. Yes. This document was handed to me by Ralph Herbst and was signed by him, as indicated in the lower right hand corner—

Q. Is that Dr. Herbst's handwriting? A. That is his handwriting, his signature and he dated it April 19, 1949.

I then—one of the purposes I wanted to accomplish with this was to send a report, progress report, to General Tire, so I made some editorial changes in my own handwriting and the writing across the top "Progress Report" and the line (246) through my name, since it was now going to go to General Tire, it was no longer appropriate to direct it to me, and so I put it from Charles C. Price and Ralph Herbst, University of Notre Dame, in my handwriting, and because I wasn't quite sure that some of the abbreviations that Herbst had used would be clear, I also changed "Brpoly" to brown polymer and "mater" I added "ial" and I added "sticky black" to complete the words he had abbreviated at the lower righthand corner, so I did make some editorial changes.

I then gave this to my secretary to retype as the document that we would send to General Tire.

- Q. Can you tell me whether Plaintiff's Exhibit 18 was understood by you to—or what it was understood to represent? A. This represented a report of experiments that Ralph Herbst had actually already finished by the date of April 19, 1949.
- Q. What about the details of the experiments? A. The first part—I guess we might say the first paragraph describes very briefly the procedure that he used to make a condensation product from 70 cc's, one mole of propylene oxide, and 6.8 grams or .05 moles of pentaery-thritol.

Q. That is the experiment that you outlined in general terms? (247) A. Oh, yes. This was work he did at my specific direction. We used as a catalyst that one and a half grams of sodium for this condensation reaction and we carried it out in a pressure vessel at 140 to 145 degrees for one sample of the polypropylene oxide glycol or polyol, which gave a yield of 85 percent of condensate, and we carried out another reaction at room temperature for 3 days, followed by heating to 110 to 115 which gave a 90 percent conversion.

So, this first paragraph describes the preparation of two different condensation products of propylene oxide

with pentaerythritol catalyzed by sodium.

The second paragraph describes some of the very preliminary experiments that Ralph Herbst did to react these polyglycol samples of the first paragraph with various isocyanates.

It has two separate headings. One in the absence of

solvent, A, and, B, in the presence of solvent.

He reports, if you look under A, he reports, polymer 1, that is the polymer made at the higher temperature reacted with IPD, which by footnote is identified as tolylene 2.4—diisocyanate or in the general nomenclature used here TDI.

The third column indicates that that polymer was also reacted with MDI. Those happen to be the same initials that have been used here in this court.

(248) The fourth column indicates that one was reacted with OTD, which is identified in the footnote as octadecyl-isocyanate.

The item in the first column 2-

Q. That is a monoisocyanate? A. That is a monoisocyanate and would not be expected to be able to give a network structure.

Then, also under A, the second sample of the one prepared at lower temperature was similarly treated. I would only call attention that under column MDI he,

apparently—he reports on two experiments, one of which he used a large excess of MDI and then in B he does the same general kind of experiments except he used a solvent in addition to the glycol.

He used as solvents actetone, alphachloronaphthalene and diethylphthalate and he reports that in the latter two cases he got rubbery materials and it was these samples which were the ones which we were happily bouncing around on the table of my desk at Notre Dame.

Q. Dr. Price, I show you a typed progress report-

Mr. Kelton: For Mr. Sweeney's benefit, I tell you this is production No. 12.

Q. (Continuing) And I ask you what this is? A. This is, in fact, the copy which my secretary typed off as a result of my notations on the Herbst (249) report to me.

It has one pencilled correction on it, which was, in fact, done by me. She mistyped April 19th as April 16th and I corrected that on the original copy before it was sent to General Tire.

Mr. Kelton: I offer in evidence as Plaintiff's Exhibit 19 the typed progress report that the witness has just identified.

Mr. Sweeney: No objection, your Honor.

The Court: It will be received.

(Plaintiff's Exhibit 19 received in evidence.)

Mr. Kelton: I don't know, your Honor, about whether your copies are legible.

The Court: They are fine.

Q. Dr. Price, after Mr. Herbst obtained these rubber balls or rubber pieces that you bounced, you said that you then asked him to prepare a larger amount. As I recall, you said it might have been the size of a small tennis ball.

Were those prepared in a different or the same way that the bouncing samples had been prepared prior to April 19, 1949 when you gave the first progress report? A. They were prepared the way-by the same procedure as is indicated for the samples in the April 19th report except that much larger quantities were used. The same (250) reagents were used and the same solvent.

Q. Did Dr. Herbst work on this project continue on other aspects of it, too, after your first progress report? Those are Plaintiff's Exhibits 18 and 19. A. Yes, he handed in at least 2 or 3-I think a total of 3 reports on the work that he did and I guess there was one in June and probably one in August and then a final wrapup in October that was submitted to General Tire.

Q. I show you a document, dated May 16, 1949, and ask you if you can identify that? A. Yes, that was the second report. It was May rather than June that Herbst handed in his second report to me.

This is a three page document signed on the last

page by Ralph Herbst.

It describes some other experiments to prepare propylene oxide pentaerythritol condensates by different procedures than those disclosed in the first report.

(251) Q. Dr. Price, I understand you to have said that this basically is a report submitted to you by Dr. Herbst? A. That is correct.

Q. Whose handwriting is at the top, namely, in crossing out "Dr. C. C. Price" and writing in "From" and what follows? A. That is my handwriting. This report from Herbst was handled in exactly the same way as the earlier one. It was retyped by my secretary with the minor editorial changes at the head before it was submitted to General Tire.

Q. What, Dr., was your practice in directing people in the position of Dr. Herbst to submit progress reports? I notice on this progress report you have stated that it is a different subject matter. A. I didn't have any usual procedure, sir. With my doctoral students, I was very informal about reporting to me and it was usually only done verbally, but because we thought this was a pretty interesting and exciting project, I did have these reports handed in to me so that this was certainly a new experience to have more or less monthly reports handed in because of the excitement that we felt about this project.

Q. For example, you have testified that Dr. Herbst (252) went along probably a few weeks after the report, which is Plaintiff's Exhibits 18 and 19, to make a larger

quantity of the rubbery bouncable material.

I don't find anything of that in there. A. That is right. We have no written report of the larger sample that was made by the same procedure, but we were very much disturbed in that first report that Herbst handed in that we got no rubbery characteristics for the samples that were made without the solvent present. Those were reported as hard, brittle materials.

It is a well known fact in rubber technology that the more tightly you bind a network, the harder the substance will be. This is the way conventional hard rubber

is made, by a high degree of cross linking.

So, we suspected that we had too many cross links in that material to make a good rubber and so it was my idea, which I outlined to Herbst, that we ought to try some other conditions for making the polypropylene oxide glycol condensation product and in this report he does report on experiments in which we tried two other different procedures for making that pentaerythirtol propylene oxide condensation product. One was to use an entirely different catalyst, sulphuric acid instead of sodium as a catalyst.

The second was to use a much smaller ratio of (253) pentaerythritol since pentaerythritol becomes the chain branches, we thought if we cut down on it we might get a looser network, so the experiment identified in the first paragraph as B was outlined by me to Herbst in order to try to determine whether by cutting down the amount of pentaerythritol we could make a significant improvement in the rubbery characteristics, since we were looking for rubber.

Hard resins are fine, but that was not the object of our search at this time.

(254) Q. In this case, doctor, in the case of Plaintiff's Exhibit 20, was there also a typed report that went to General Tire? A. Yes, sir.

Q. I show you a document headed "Progress Report," dated May 16, 1949. A. Yes. This is the typed off copy which has now been condensed enough to appear on two pages rather than 3.

Mr. Kelton: T offer in evidence as Plaintiff's Exhibit 21 a document headed "Progress Report," May 16, 1949, which the witness has just identified.

Mr. Sweeney: No objection, your Honor. (Plaintiff's Exhibit 21 received in evidence.)

Q. I show you a document, dated August 8, 1949, and ask you if you can identify that. A. Yes, sir. That is the third of the progress reports handed to me by Ralph Herbst. It is signed and dated by him on page 3 and then has a considerable amount of handwriting in my writing on it in addition.

Mr. Kelton: I offer it in evidence as Plaintiff's Exhibit 22.

Mr. Sweeney: No objection. (Plaintiff's Exhibit 22 received in evidence.)

Q. With regard to Plaintiff's Exhibit 22, you have (255) indicated there was considerable handwriting on the first page of the document. Can you tell me whose it is and what it is? A. All the extra handwriting on the first page is mine, including that up at the very top of the page, "G. H. Swart".

Q. What was the occasion of putting G. H. Swart on there? A. These reports were being typed to be sent

to him at General Tire.

Q. What aspect of your work did this report pertain to? A. This report again begins by describing some additional detailed procedures for making the propylene oxide pentaerythritol condensates. They are abbreviated by now as PO-PET, which is propylene oxide pentaerythritol.

Several different procedures for making this condensate product are reported and then we report on the removal of the sodium from some samples which had been made by sodium.

In our early experiments, we used sodium as a catalyst and did not remove the sodium before we went on to make the condensation.

For several reasons, we now wanted to remove the sodium so the term diionization, which is used in the (256) very second paragraph at the top, refers to the procedure for removing the sodium.

We then used the diionized material to measure its molecular weight in the paragraph at the middle of the page, then determined the molecular weight to be 640 average molecular weight. It was abbreviated in this report as Mol. Wt.

The other major function that we used the diionized material for was to make the polyesters which we had originally conceived. The sodium would be very detrimental in the condensation to make the polyesters, so it was essential to remove the sodium used as a catalyst

in the initial condensation before the final step of network formation by polyester and that polyester formation is summarized as paragraph 3 on page 2 of this report.

The final page merely gives some additional details

of the sodium catalyzed reactions.

- Q. As I understand it, Dr. Price, there is no diisocyanate work reported in this report. A. No, sir. There is no isocyanate work reported in here. These are entirely some reports of the second major approach, the polyester approach. It did give rubbers, but they were not very good ones, but we did report on page 2 that we did get some rubbery characteristics with these materials, but they were rather weak and not (257) nearly as exciting rubbers as the polyurchane rubbers that we made in the earlier report.
- Q. And in this case, did you render a retyped report to General Tire? A. Yes, sir.
- Q. I show you a paper headed "Progress Report," which bears on the lower lefthand corner of the last page "Notre Dame, Indiana, August 8, 1949," and I ask you if you can identify that for me. A. Yes, sir. This is the typed off copy with the handwritten changes on 22 now typed on 23.
- (269) Mr. Kelton: Your Honor, I also offer in evidence Plaintiff's Exhibit 15 for identification. I understand I haven't done that and that was an oversight.

Mr. Sweeney: This was one of the ones I objected to and your Honor took it for the limited purpose yesterday.

Mr. Kelton: This, of course, is offered for the limited purpose which was stated yesterday.

The Court: You hadn't offered it yesterday.
Mr. Kelton: I had not offered it. I just omitted it.

The Court: It is admitted subject to Mr. Sweeney's (270) reservation and it is submitted for the limited purpose as I think 12—

Mr. Sweeney: 13 and 14.

The Court: Yes.

Mr. Kelton: For that purpose, your Honor.

(272) Direct Examination by Mr. Kelton:

Q. Dr. Price, yesterday in answer to a question, which asked you how you proceeded with Ralph Herbst in connection with this project that you had in mind, among other things you said:

"The basic idea, which I outlined to him, was to make polypropylene oxide with three or four hydroxl groups to serve as cross links for the network. To react such glycols which we would have to make ourselves."

Now, I ask you what were you referring to in the term "such glycols"? A. I was referring to the immediate preceding phrase, which was the polypropylene oxides made with three or four hydroxyl groups, which we actually made by condensing propylene oxide with pentaerythritol.

Q. Now, the only problem I had, I think for purposes of this case we have taken glycol or ethylene glycol or propylene glycol as being a hydroxyl compound, that is difunctional, and did you use glycols in that term there? A. No. I specifically referred there to a polymer which had four hydroxyl groups.

The polymer, however, can be considered to be a polymer of propylene glycol and this whole industry (273) is frequently referred to as a polyglycol industry because the polymers can be construed to be polymers of glycols and I used the term in there as it is used generically in the industry as a polyglycol.

Q. The part I just referred to of your testimony was on page 234 of the record.

Over at page 247 at about line 6 you referred to "poly-propylene oxide glycol."

Can you clarify that? A. May I see the context in which

I used the term there, Mr. Kelton?

Q. Yes. This statement again was in reference to your work. A. This again refers to the sample of the condensation product of propylene oxide with pentaery-thritol and clearly is a polymer which contains four hydroxyl groups.

(274) Q. Dr. Price, when we concluded yesterday afternoon, we were discussing Plaintiff's Exhibit 23, which was a progress report dated August 8, 1949. A. Yes, sir.

Q. Did you submit that report to General Tire? A.

Yes, that was submitted to Mr. Swart.

- Q. About when? A. Within a few days of the date. I would assume no more than a week of the date of August S, 1949.
- Q. Did you make any progress reports subsequent to this one of August 8, 1949? A. There was one more report that was made to General Tire, which was both a progress report on work done subsequent to August 8 and a supplement to the entire set of reports, supplementary to all 3 progress reports.

This report was submitted to me about the first of September and, as I recall, was forwarded on to General

Tire in October of 1949.

Q. I show you a copy of a paper headed "Summary Report," and ask you if you can identify that. A. Yes. This is the report that I just referred to. This is the report as submitted to me by Ralph Herbst and signed on the fourth page, Ralph L. Herbst, Jr., by him.

It does not contain a date by him, but this is the (275) report submitted to me, I would guess, in early

September of 1949 by Dr. Herbst.

Q. Let me ask you if you can identify any of the hand-writing and the modifications. A. Yes, sir. As had been my custom with the other progress reports, this report was altered to be typed for submission to General Tire, so the "to" was changed from my name to General Tire and Rubber Co.

In my handwriting in the upper righthand corner it says 3 cc's, which means 3 carbon copies. "From the University of Notre Dame," that is in my handwriting.

I also deleted the 1, 2 in parenthesis after Propylene oxide, since it was obvious that this was, in fact, the propylene oxide we were using.

I added a change on page 2 in my handwriting, changing "in order to obtain" to "in the hope of obtaining".

On the last page, I added my signature under that of Ralph Herbst and added, "Notre Dame, Indiana, 24 Octoher 1949," which is when I had gotten around to preparing this document for submission to General Tire.

- Q. When you say "preparing the document," what do you mean by "preparing"? A. Making these minor editorial changes to instruct my secretary how to type the document up for submission to (276) General Tire.
- Q. In its typed form, before amendment, where did it come from? What do you know about it? A. The unamended—the document, without my handwriting on it, was presented to me by Ralph Herbst as a report of the experiments he had done in the last three weeks of August and as supplement to some of the earlier reports he had presented during the course of our project.

Mr. Kelton: I offer in evidence as Plaintiff's Exhibit 24 the document which the witness has just identified.

Mr. Sweeney: No objection, your Honor. (Plaintiff's Exhibit 24 received in evidence.)

Q. Dr. Price, this was retyped and sent to General Tire about when? A. Very shortly after that date of Octo-

ber 24, 1949. I believe in this case I did submit this to Mr. Swart with a cover letter, so I could get the exact date from the cover letter by which that was submitted to Mr. Swart.

(277) Q. I would like to go back, Dr. Price, to Plaintiff's Exhibit 18.

Dr. Price in view of our previous discussions about glycols and your uses of the term of a propylene glycol to include the condensate of propylene oxide with pentaerythritol, I would like to go over on the first progress report, which is Plaintiff's Exhibit 18, this and I would ask you to tell me whether this involved the production of a condensate of propylene oxide with a polyhydric alcohol containing 3 to 4 hydroxyl groups.

By three to four, I mean either 3 or 4. A. Yes. The experimental conditions outlined there specify that we condensed pentaerythritol, which is an alcohol (278) containing four hydroxyl groups, with 20 equivalents of propylene oxide, so that the product formed in the first step, that is, as described in paragraph 1, is a condensation product of a tetrahydroxyl alcohol with propylene oxide.

Q. After that condensate was in hand and, as I understand, there were two condensates, in other words, made in two ways— A. That's correct.

Q. One was made by a pressure reaction at an elevated temperature and the other was made by reaction at room temperature for three days, followed by heating. A. That is correct, but the reactants were the same in those two different preparations.

Q. Right. Now, doctor, having the condensate in hand, what was done with it? A. The second step on our planned preparation of polypropylene oxide rubber was then to condense this liquid condensate with disocyanates and the two disocyanates that were used were MDI and a product now referred to as TDI, but identified in this report as IPD.

Q. What about the ratio of isocyanate to the hydroxyl groups or active hydrogens? A. In order to make a network structure, which is what we envisaged, it was essential to have—it was absolutely (279) essential not to have an excess of hydroxyl groups. If there were an excess of hydroxyl groups in the condensation with the diisocyanate, there would be no way we could get an indefinite network structure, for which we were aiming.

So, our procedure was to add enough of the diisocyanate to be sure that we exceeded the ratio of one to one, because if we had an excess of isocyanate groups, we could get a network structure; if we had an excess of

hydroxyl groups, we could not.

Q. When you say the ratio of one to one, are you talking in terms of weight to weight, volume to volume or some other thing? A. This is talking in the chemist's term of mol to mol, reactive group to reactive group.

The Court: Are you about to leave 18, because I want to ask a question before you do.

Mr. Kelton: I would be pleased to have you ask it, your Honor.

The Court: All right.

By the Court:

Q. Dr. Price, obviously I don't know what I ought to understand, so I am going to find out about this:

Reading 18, what am T to understand about the last line in the paragraph, or the last sentence in the paragraph (280) under 2, which reads—I guess that is—".5 grams of unreacted pentaerythritol was recovered from one."

What does that mean, "was recovered"? What am I to understand by that? A. This sentence has to do only with the very first step in the process, your Honor. It has to do with the preparation of the condensation product of pentaerythritol with propylene oxide.

By the procedure 1, when we looked at the product, we found there was still some solid, unreacted pentaery-thritol, which we could recover from the liquid condensation product.

In the case of the procedure 2, all of the pentaerythritol which we put into the reaction mixture was converted to liquid products.

Q. I see. A. So, this had to do solely with the first step, the preparation of the liquid condensation product which was then used. In one case, we recovered a half a gram out of one and a half grams of pentaerythritol remaining unreacted and was unrecovered and in the other case we recovered none.

By Mr. Kelton:

Q. Dr. Price, there is, on Plaintiff's Exhibit 18 and Plaintiff's Exhibit 19, the retyped one that went to General Tire, an indication which on Exhibit 19, the retyped portion, (281) is hard brown polymer, hard black polymer.

Under the IPD column and the MDI column with regard to one of the condensates, the propylene oxide pentaerythritol condensates that was used—can you tell me, did you see those products at the time? A. Yes, sir.

Q. Did you make them, yourself? A. Oh, no. These were all done in the laboratory by Dr. Herbst.

Q. Did you have any conclusions about whether you had made a polymer in the case of the hard brown polymer and the hard black polymer? A. Absolutely no question that we had. The starting material, as I said, was to be a liquid. The fact that it was converted to a hard brown or hard black substance was a clear indication that we had very substantially increased the molecular weight of this material and, in fact, we are convinced that we had made a network structure in these hard brown and hard black polymeric substances.

Q. Did you envision that they would be useful or useless at the time? A. Well, there are many uses to which such hard brown or hard black polymers could be used. Bakelite, for example, is exactly such a polymer but we, of course, in this project, (282) were mainly aiming at trying to make rubber, so these had uses but not as rubbery composition.

They were much too hard to be construed as elastomeric or rubbery.

Q. Now, in the case of the column OID, which you said yesterday was octadecylisocyanate, were you surprised or not surprised that you got only a waxy solid? A. No. We were not surprised at that. We were quite certain we should not get a network structure and the properties of this material indicated it was not.

The octadecyl group is an important constituent of many waxes, so it is not at all surprising that a material which comprised octadecylisocyanate should have a waxy feel, but these were some of the waxy materials and not hard, impervious polymers.

Q. You have said that you didn't expect to get a network with the octadecylisocyanate. Will you tell me why that is true? A. Octadecylisocyanate has only one cyanate functional group. It could react at the hydroxyl groups of our condensate and chemically convert it into a new derivative, which would have octadecyl groups at the end of the chains where a hydroxyl group had been, but the octadecyl group then had no way to link with other chains. So, there was no (283) possibility that this could have or give a network structure and we wanted to do this experiment to distinguish between the diisocyanate, which would give a network structure, and a monoisocyanate, which would not give a network structure.

Q. Now, Dr. Price, following this or with respect to the so-called summary report, you considered that the caption "summary" correctly describes the report? A.

No, it is not best described as a summary report, because we did not, in fact, summarize all the information from the earlier progress reports.

It would be much more accurately described as, (a) a progress report in which we reported the progress from the time of the October 8th, I believe, report until Dr. Herbst finished his experiments, so that part reported some new experiments, which had not been reported in the earlier progress reports.

The other part of the report was, in fact, a supplement to the earlier reports in giving some of the details which Dr. Herbst had not included in the earlier brief progress reports.

Q. Following the submission of the last report—let me ask you, were there any further reports from Mr. Herbst on this work? A. No, sir. No, he left to go to Utica College, to (284) his teaching assignment and did no further work to my knowledge on this subject.

Q. Did you do anything further in connection with this matter of polyurethane polymers from the condensate of propylene oxide with a polyhydric alcohol containing 3 to 4 hydroxyl groups and then reacting that with a polyisocyanate? A. Well, I did two other things, myself.

One was to instigate some further research work in my own laboratory in an effort to make higher molecular weight polyglycols, if I might use that term again.

We were convinced that we could improve or get a more extensive range of rubbery properties if we could improve the art of making propylene oxide to get longer molecular weight chains.

Leon St. Pierre did a PhD thesis in my laboratory on some very extensive further work on the polymerization of propylene oxide. In fact, we made some patent applications on his work.

In addition to the work we did in our own laboratory, I continued to stimulate the General Tire people to evaluate the practical applications of our discovery and to get the—

- Q. Now, doctor, you have referred to the St. Pierre work, I think it was. (285) A. Yes.
- Q. That has to do, if I understand you correctly, with trying to extend the length of the chain of these polypropylene oxides or polyglycols, I guess you call them. A. That is correct.
- Q. That, as I understand it, was the attempted improvement in one of the reactants that could be used in the work that Dr. Herbst did. A. That's correct.
- Q. When you say "our discovery," I am not quite sure what you mean.

The Court: Would you illuminate a little more, because I think I was about to get a little confused here.

When you say "extending the chain," you are not therefore making a distinction that Dr. Mark made yesterday about a linear or network, are you?

The Witness: No, sir, your Honor. I am talking about the preparation of the preliminary condensation product. We did do one molecular weight measurement that is reported in our reports on the condensation product of propylene oxide with pentaerythritol and it was something of the order of magnitude of 640.

We were sure that—I was sure; I use "we" sometimes when I shouldn't technically use it—I was sure that (286) it would be interesting if we could also make longer, higher molecular weight glycols as the starting material, polymers, the preliminary condensation product, should be made if we could possibly discover ways to do it, of higher molecular weight.

It was this stage in the reaction which Leon St. Pierre was exclusively involved in. He did no other work that I can recall in the second stage, the extension into the network structure.

Does that clarify that? The Court: I think so.

Q. Would this be of any help, and I am referring to Exhibit 4H? A. It might be possible to use that to indicate the nature of our concern.

The chart indicates in a schematic way the condensation product in this case of glycerol, a trihydric alcohol with propylene oxide. It does not indicate the number of units of propylene oxide. Those are left indefinite.

In one example, we proved that the total weight of this was only 640. That limited the length of these polypropylene oxide units that had grown on the initial glycol. We wanted to see if we could make those arms longer.

Q. Now, can we get back to what you did subsequently (287) with regard to the work that Dr. Herbst did under your supervision from March through August or into early September of 1949? A. I visited the General Tire Company fairly regularly, probably on the order of magnitude of six times a year, for a day or two at a time, to discuss various chemical problems with their research staff.

I continued to stimulate them to do work on this problem. As a university laboratory, we were not in a good position to do what I would call the development work on this invention—

Q. Doctor, may I ask you to what you refer when you say "this problem"? A. On this invention, on the development of a commercially useful propylene oxide rubber, which was the invention that I had and which Herbst accomplished in April of 1949, so the commercial application would have to be done, not by Notre Dame; we were not a commercial organization.

It would have to be done by General Tire.

In view of the fact that my arrangement with Notre Dame was that any discovery made would be assigned to the University of Notre Dame, in view of the fact that all the work that was done there was done under a contract between (288) the University and General Tire, General Tire had the rights to an exclusive license to any inventions that came out of the work done under this General Tire project,

So, its commercialization should be done by—must be done under the terms of that arrangement by General Tire. So, one of the activities in my consulting visits to General was to continue to stimulate them to move this work along in two directions:

First, to do further development work to see what the commercial applications might be and, secondly, to get a patent filed because, again, part of our agreement—our agreement being mine with the University and the University with General Tire—was that they would undertake the filing of patent applications so the patent application was to be handled by the General Tire Company.

So, it was part of my activity to stimulate them both on the commercial development and on the securing of a patent application.

- Q. Did you ever have any discussions in the period 1949 through 1953, let us say, with a T. A. TeGrotenhuis? A. Yes, sir. He was a patent advisor or patent counsel to General Tire.
- Q. Did you have any occasion to talk with Mr. TeGrotenhuis with regard to speeding up this thing or lighting (289) a fire under him? A. Yes, I am sure I had a number of occasions to talk to him and I think finally, in 1953, I became aware that DuPont had some patent applications on polypropylene oxide rubber and I think sometime in 1953, I wrote to him to again urge him vigorously to get our patent application or my patent application, I should say, on file.
- Q. I show you a letter, dated May 15, 1953, and ask you whether that is the letter that you referred to. A. This is a letter on the University of Notre Dame, Department of Chemistry, stationery, dated May 15, 1953, from me to Mr. T. A. TeGrotenhuis, Evans & McCoy, Bulkley Building, Cleveland, Ohio. He was in private practice in Cleveland, but was retained by General Tire.

This letter does in fact indicate that I had heard disturbing to me, very disturbing to me, news that there were some DuPont applications in the general field of propylene oxide rubber. It mentions that I had in fact done this back in 1949 and thought we could get this together.

I ended up by asking him what he thought of the urgency of getting my patent application filed.

(290) Mr. Kelton: I offer in evidence Plaintiff's Exhibit 26, the letter of May 15. 1953, which the witness has just discussed.

Mr. Sweeney: No objection, your Honor.

The Court: Dr. Price, in regard to Plaintiff's Exhibit 26, I see a new term in here that I haven't seen before and I would appreciate it if you would explain it to me.

In the third paragraph "maleate' styrene" appears in the first sentence of that paragraph. What does that mean? That is a new term for me.

The Witness: This, your Honor, is a new term perhaps. This refers to the polyester extended material, the maleate was one of the dicarboxylic acids which we used to extend the chain in some of those reports of Herbst. You recall we did the disocyanate extension, we did the dicarboxylic acid extension.

The isocyanate words refer to that chain extension. The maleate refers to the dicarboxylic acid chain reaction and was, of course, not part of the final issued claims in the patent.

By Mr. Kelton:

Q. Dr. Price, in the second paragraph of the letter, in the last few lines of that paragraph you say: (291)

"There is also evidence that the idea was disclosed to a varied group at a QM conference in Washington on February 1, 1949."

What I would like to know is what idea are you talking about? Are you talking about the invention? A. No, sir.

Q. As you were trying to get it patented? A. No, sir. The idea that was discussed at the—QM, incidentally, stands for Quartermaster Corps—the idea was discussed at this conference was the general broad idea of making a network of propylene oxide backbone.

The Court: That was the conference you referred to that led to the Herbst study initially?

The Witness: That is correct, your Honor. But at that time the idea was just the very broad, general principle that it would be desirable to make a propylene oxide network. It was not until I got back to Notre Dame that the practical procedure for making it through these chain extensions became obvious as the way to try to accomplish that objective after having tooked at the state of the art.

- Q. It is what you testified to earlier in connection with this Quartermaster Corps conference? A. That is correct. That is exactly correct. I think (292) I only wanted to mention this here to indicate that other people might have gotten interested in this idea and to in the last few lines of that paragraph you say: (292) I only wanted to mention this here to indicate that other people might have gotten interested in this idea and to light a little fire under the attorney to get our case filed.
- Q. Can you tell me whether any work was going on at General Tire, so far as you know, to explore the limits of your invention or anything else? A. Yes. General Tire did undertake work in their laboratory to explore the possibilities of commercialization of a polyether polyurethane rubber. They eventually became one of the producers of the rubber.
- Q. Dr. Price, can you tell me whether or not you were successful in filing a patent application? A. Yes, sir. As a result of my endeavors, some time a few months after this letter to Mr. TeGrotenhuis, a patent application was filed in my name. I don't remember the exact data but it was in the fall of 1953.

Q. I show you Defendant's Exhibit A and ask you if the patent application, which was filed, eventuated in the issuance of your patent No. 2,866,774. A. Yes, sir.

Q. I call your attention to the fact that this application for that patent was filed September 23, 1953. A. That is correct. That is about four months after (293) my letter to Mr. TeGrotenhuis.

Q. I note from Defendant's Exhibit A that the patent issued on December 30, 1958? A. Yes, sir.

Q. Did anything happen in connection with the patent after its issuance? A. It certainly did.

Q. What was it, briefly? A. I don't remember the exact date, but some time shortly after its issuance—by "shortly" I mean some months—an interference was declared between my issued patent and an application that was on file in the name of Saunders and Heiss. This interference proceeding took several years—I guess nearly four years to be resolved, but we won the interference and got the clear right, I think it was, in 1962.

Q. Did you testify in that interference? A. Yes, sir.

(296) Cross Examination by Mr. Sweeney:

Q. Dr. Price, where do the copies of the reports which have been marked here in evidence come from? A. The copies came from my files.

Q. That would have been when you were at the University of Pennsylvania, your files there or your files at Notre Dame? A. These files were first put together at the time of the interference proceeding. Yes, when I was at the University of Pennsylvania.

Q. Did you search the files at General Tire for the

corresponding documents? A. No, I did not.

Q. Do you know whether or not such a search was made? A. I might make one point: That the progress reports that were presented here from Ralph Herbst were the originals. They were never sent to General Tire. They (297) were his originals that were given to me.

It was only the retyped copies of those documents which I retained a carbon copy of and the originals of which were sent to Mr. Swart at General Tire.

So, there are some of the documents that have been presented here that are original copies. Xerox copies of original signed copies and some are Xerox copies of my carbon copies in my files.

Q. My question was do you know whether anybody looked in the files of General Tire for reports you sent them? A. I don't know the answer to that question.

Q. May I ask you to look at Plaintiff's Exhibit 18.

Mr. Sweeney: If you don't mind, I would just like to put the exhibits in front of the witness, your Honor. I think it would make it easier.

The Court: Surely.

Q. This is Dr. Herbst's first report to you; is that correct? A. Yes; that is correct.

The Court: This is Plaintiff's Exhibit 18 you are talking about?

Mr. Sweeney: Yes, your Honor, Plaintiff's Exhibit 18.

(298) Q. You have testified that the reaction that is specified under 1 and 2, which are alleged to be a reaction between propylene oxide pentaerythritol gave you a condensate. Was that condensate ever analysed? A. You are referring to the condensate that was made in the first paragraph?

Q. Well, there are two of them. I thought I would cover them both together. Were they ever analysed, Dr. Price? A. By that you mean elementary analysis?

Q. To find out if they were, in fact, the condensate you thought they were? A. I am not sure I understand what kind of analysis you are referring to? They were not subject to an elementary analysis, which is what I would understand your question to mean.

Q. I am not sure that I understand what you mean by elementary analysis? A. Analysis for carbon, hydrogen

and oxygen; analysis for the elements. This is what one normally does when one makes a new compound that one wants to report in the literature and to me, as an organic chemist, when you ask me if I did an analysis of a substance, it would be an analysis for the percent carbon, percent (299) hydrogen, percent oxygen, percent chlorine that might be present. We did not do that for these compounds.

Q. Did you do any kind of analysis of this material, Dr. Price? A. Yes, we converted it to the network

structure by further reaction.

Q. But you didn't analyse the so-called condensate to find out if it was, in fact, the condensate you thought it was? A. There is no question it is a condensate. Of that there is no question because the original material, one was a solid, hard, solid, soluble; the other is a liquid that boils at 35 degrees and would evaporate completely.

These were viscous fluids which did not evaporate so they were a reaction product. They were not the start-

ing materials. That is unequivocal.

Q. Okay. What I am talking about is the reaction product? A. Yes, that is what I am talking about.

- Q. I am asking you whether you analysed the reaction product to find out what it was? A. We knew it was a condensation product of pentaerythritol and propylene oxide.
- (300) Q. But you did not analyze it? A. Well, we analyzed it in the sense that we looked at the product and we knew it was not the starting materials. Therefore, it had to be a reaction product.

Q. Just what that reaction product— A. In my view

that is a form of analysis, Mr. Sweeney.

Q. All right, Doctor, I don't choose to argue with you. You testified earlier today about the excess of isocyanate question and with reference to this exhibit I believe you said you had to have an excess to get the network that you wanted to get. A. That is correct.

Q. Does the report say anything about an excess? A. In one place under MDI, it does say that in one

particular example we used an excess of MDI and in that particular case we used a gross excess of MDI.

Q. That is the only reference to excess, though? A. In

this report, ves.

- Q. The rubbery material that you talked about from which the rubber balls came, is the rubbery material under the section headed "In presence of solvents, acetone, alphachloronaphthalene and diethylphthalate." A. I am not sure I understand your question.
- Q. You testified that there were some odd shaped rubber (301) balls brought to you. A. Yes.
- Q. Those came from the rubbery material which is listed as being reacted in alphachloronaphthalene and diethylphthalate. A. That is correct.
- Q. That is the same material as this so-called later

tennis ball was made from? A. That is correct.

- Q. There isn't any report on the tennis ball? A. There is no report in the progress report of that; that is correct.
- Q. If you would, then, look at Exhibit-I think before I leave that Exhibit 18, Doctor, I would like to ask you a couple more questions.

Do you consider any particular reaction as shown in this report to be a successful reaction? A. Certainly.

Q. Which one? A. All of them. They all gave a reaction, a polymer network by the condensation of our condensate with the diisocyanates. So they all were successful reactions.

We were hoping to make—we had hopes in addition to these successes to also make a rubber without the solvent (302) present. That was one of the objectives which we did not-had not achieved at the time of this progress report.

Q. I gather, then, that you were particularly pleased with the rubbery materials that you got? A. Since we were working for General Tire & Rubber Company, in-

deed, yes.

Q. Now, if you will look at Plaintiff's Exhibit 20, which I think is within the pile that you have— A. Yes, I have it, Mr. Sweeney.

Q.—this report also refers to the reaction with diioscyanate compounds. I think if you will look at page 2 at C there is a reference there to the reaction with

diiosocyanate compounds. A. Yes, sir.

Q. Is there any indication in that section of the report about the excess of dilosocyanate? A. Only by implication in the statement at the bottom that the material was insoluble and the only way to have a material which was insoluble in organic solvents was for it to be a network structure of some kind. So, it was not stated directly, only by implication.

The Court: I am sorry, sir. When you say the bottom— The Witness: The bottom of page 2 of Exhibit 20, (303) the last line. "All attempts to dissolve this rubbery material in organic solvents failed."

The Court: Fine.

Q. These two reports, then, seem to indicate that Doctor Herbst did some work in connection with reacting the disocyanate with the polyol. Is there any other indication in your reports of actual tests that Doctor Herbst ran on the disocyanate polyol reaction? A. I have some recollection in the last report he made some allusion to it. Maybe I should refresh my memory.

Q. Please do. A. It is Exhibit—the summary report it is Plaintiff's Exhibit 24 on page 3. He refers to reactions between the condensate and diisocyanates, and he describes in considerably more detail the procedure

he used for that condensation.

He does give some properties—he describes the procedure as based on the fact that the initial reaction of the condensate with disocyanate was a vigorous reaction. It evolved heat.

So that he added the diisocyanate until that vigorous heat reaction was over, which we took to be the case when he had reacted all the hydroxyl groups with the

diisocyanates. (304) He then added a little more diisocyanate compound to make sure we had an excess and then warmed it, and he does describe a few of the properties of some of the diioscyanate products at that point.

He describes some as hard, brittle polymers. The octadecyl isocyanate produced viscous oils, he mentions at this time, with some of these condensations.

He also describes there that when the reaction was used in a solvent, as in the first report when he used the alphachloronaphthalene, it generally required a higher temperature for the condensation reaction to occur.

Q. You said that this section of the report says so, that he added more isocyanate to get an excess. Where does it say that? A. Again the words "excess" are not there. He added diioscyanate as long as there was a vigorous reaction occurring drop by drop. You can feel the heat evolve.

When this amount of diiosocyanate had been added, we assume we had the reacted hydroxyl groups. To be sure, if we had an excess, we then added a few more drops. It does not say that is the purpose of it, Mr. Sweeney, but that is the reason that he did it in that particular way.

- Q. All T was trying to establish, Doctor, is that it doesn't say adding an excess in that portion of the report. (305) A. We did not specifically say so; you are correct.
- Q. Do you believe that this section of the report, which is plaintiff's Exhibit 24, is a description of an additional reaction or a description or a summary of what he has done in the other two reports? A. This is a description of the procedures that he had used earlier to make the diisocyanate condensation products, and I don't believe there is any indication that this was a new experiment.
- Q. That is what I thought. I just wanted to clarify that, Doctor.

He says in the last part of that paragraph that only slight improvement resulted in the rubbery properties of the polymer. This is when he is carrying it out in a solvent. What does that mean? A. I guess you will have to—I don't know the answer to that, exactly what Doctor Herbst had in his mind when he wrote that sentence.

Q. Would you look again at Plaintiff's Exhibit 20? A. Yes, sir.

Q. On page 2 of that exhibit under the paragraph marked b, where it says "using propylene oxide"—strike the question. I have got the wrong place. Just a minute.

Would you look, then, at Plaintiff's Exhibit 24 (306) again, Doctor. On page 2 you changed "In order to obtain" to "in the hope of," and I am just curious as to why you did that. You have been so positive about having obtained these materials that I wondered why the "in the hope of" was put in there. A. May I read the sentence?

Q. Yes. A. "The low catalyst condensations were used in order to obtain high molecular weight polymers."

The fact is that that was our hope, but that was not our realization. We did not get higher molecular weight when we used a low catalyst concentration as to higher catalyst concentrations. We were puzzled by that, and finally learned an answer to that, but it was because, in fact, we did not obtain what we hoped that I thought we ought to change the wording from "in order to" to "in the hope of."

Q. Look, then, if you will, at the conclusions on page 4 of Plaintiff's Exhibit 24. That paragraph would indicate that the best rubber products that you got out of this research program were made using the acid anhydrides so that you got polyesters, wouldn't it? A. That was certainly true in the absence of solvents; that is absolutely correct. In the absence of solvents (307) the diiosocyanates gave only hard, brittle materials.

Whereas in the absence of solvents, the esters gave rubbery products.

Q. Now I am confused. You were working for General Tire who was interested in rubber. You were interested in rubber. This was supposedly the whole reason for starting this program, was to find another kind of rubbery material.

If you got satisfactory rubbery materials in solvents, why doesn't the conclusion under this final report say something about them? A. This report was written by Doctor Herbst. I don't know why he didn't mention it, but certainly in my mind there was no question that we had made a rubber from the diisocyanates, and we reported that we had made a rubber, and we did make it, and we took samples of it to General Tire in the presence of the alphachloronaphthalene.

The only further fault that I can add to this is that in general in polymers when you add a plasticizer or a solvent, you weaken the polymer, and so it would, of course, be desirable to be able to make the rubber with-

out that diluent present in the rubber.

Q. I understand that. What I am curious about is why this conclusion of your final report, which maybe you didn't write, but you signed and sent on says only that the (308) reactions with disocyanates produced hard, brittle polymers. A. That is the situation in the absence of the solvent, of course.

Q. You did not say anything about that. A. Not in

this particular sentence, no.

Mr. Sweeney: I will ask the clerk to mark this as Defendant's Exhibit B.

(Defendant's Exhibit B marked for identification.)

Mr. Sweeney: I might explain that this, your Honor, is a type-off of a letter, which was supplied

to us by counsel for General Tire. The letter was almost illegible.

The Court: All right.

Q. Doctor Price, I will hand you Defendant's Exhibit B, which is a letter dated June 17, 1949, written by you to Mr. T. A. Tegrotenhuis. I believe you identified that during your deposition.

That letter was written after Doctor Herbst had done his original work as reported in Exhibits 18 and 20, I believe. I would like you to look at the second page, the first full paragraph, which begins, "vulcanization."

What do you say about the use of disocyanate as a cross linker in this material in that paragraph? A. When disocyanate was used as a cross linker, (309) the material formed a hard, brittle—

Q. You didn't say anything about rubbery there either. A. No, this was because this was without the diluent present, without solvents.

Q. And, yet, when you wrote the letter you started out by saying that you wanted to tell him about the preparation of vulcanized rubbery polymers. A. Right.

Q. But you didn't tell him about how to make a rubbery polymer using a solvent? A. That had already been disclosed to him in the progress report fr in Doctor Herbst.

Q. Did Mr. Tegrotenhuis get the progress reports? A. I guess I shouldn't be able to say that. They went to Mr. Swart at General Tire and I don't know for certain that Mr. Tegrotenhuis saw the copies of the reports, although I would assume it would be reasonable that he did.

Q. You testified yesterday that this program, if you will, came to you in January or thereabouts in 1949, and that you knew nothing about polyethers at the time. I believe that at about the same time in your testimony you said you were aware of the German work at Bayer.

Can I take that to mean that you were aware of the work that was in Doctor Bayer's report that has been marked here as an (310) exhibit? A. I wouldn't be certain that I was aware of this paper, but I was certainly aware of the fact that Bayer and his company had developed the polyester urethane rubbers.

Q. Have you ever read his paper, which is Plaintiff's

Exhibit 7-A and B. I believe? A. Yes.

Q. Do you think it discloses the making of the propulene oxide rubbers as you made them? A. No.

Q. Does it suggest doing what you did? A. Well, I have a little trouble with the word "suggest." It certainly suggests that you can make network structures by extending polyhydroxy compounds with diisocyanates. The thing he does not suggest to me is that these segments that you would like to extend should be polypropylene oxide segments.

Q. Would you look again at Plaintiff's Exhibit 24.

A. Yes, sir.

Q. In this paragraph we were discussing earlier on page 3 about the reaction of the diisocyanates, down at the bottom you mention carrying out the reaction in a solvent. Now, what was the solvent that you were referring to there? A. Well, as I said, this refers back to details of (311) work that was done in the first progress report, and in that progress report we actually reported three solvents, I think, if you will recall. I think that was in Plaintiff's Exhibit 18.

acetone as well as alphachloronaphthalene and diethylphthalate.

The acetone is a volatile material and was easy to

remove.

The other two were nonvolatile materials, they were not easily removed from the polymers,

So, I presume that that paragraph refers to all three of those solvent systems; that they were done more or less in the same general way as is described in paragraph 3 of Plaintiff's Exhibit 24.

Q. But paragraph 3 of Plaintiff's Exhibit 24 in connection with the solvent says that when the reactions were carried out in a solvent, you got rubbery properties. You didn't get rubbery properties in acetone, did you? A. That is right; we did not.

Q. What are referred to there are the alphachloronaphthalene and the diethylphthalate. A. Well, not certainly as regards the first—that sentence has got a semicolon in it that separates it into two separate ideas.

The first idea is that if you used a solvent, and (312) I would certainly interpret that to mean that even if you used acetone, to get the condensation to go, you needed a higher temperature.

The second part of the sentence refers to the properties of the products, and there it is clear that the rubbery products were obtained only with the nonvolatile solvent that we used.

Q. That is all T was trying to get established, Doctor. Am T also correct that those two solvents could be called plasticizers? A. Certainly they could.

Q. They are both high boiling liquids? A. Yes, and that is the property that would characterize them as possible plasticizers.

Q. So that the only successful rubbery polymer work that you did in this program from April of 1949 to August was in a plasticizer, am I correct? A. Could I have that question read back to me?

(Question read.)

A. No. Oh, no, we made rubbers during that time by the polyester process.

Q. You are correct and I should have put that limitation on my question. A. I thought you might.

(313) Q. What I am referring to is your urethane work.

With that limitation. A. The only rubbery urethane that

we made in Herbst's works was done with what we call the solvents.

- Q. You will agree with me again it is still a plasticizer? A. Yes.
- Q. From the reports that we have here in evidence, the only reactions that Doctor Herbst ran in making ure-thanes from this P.O. condensate are the ones listed in the first two reports, Plaintiff's Exhibits 18 and 20? A. I want to be sure I got that question correctly again. Could you reread that to me?

(Question read.)

A. The way I must answer that is that he did many other reactions, including the tennis ball, which was this kind of a reaction, so he did many others which were reported in his notebooks, but the only ones that were reported in these reports are as you have stated.

Q. Whatever happened to the tennis ball? A. That was taken by me on one of my visits to General Tire to show to Mr. Swart. What he did with it I have no idea.

Q. Do you know what happened to the little rubber balls? (314) A. The little rubber balls were in my desk for many years, in my top desk drawer where I keep my pencils, and I would occasionally take them out to play with them to see how they were surviving.

I don't know what happened to them when I left Notre

Dame.

Q. You testified today that after the final report, Plaintiff's Exhibit 24, was submitted that further research work was done in your laboratory and you mentioned Doctor St. Pierre.

Am I not correct that his work was limited to diols? A. That is correct.

Q. So that he was not working on improving the chain extender or the chain links of the propylene oxide material which you had used in your urethane program? A. He was working to improve the chain length of propylene oxide polymers, and if we could discover how to

do that, it would be a simple matter to then apply that to the chains extended on pentaerthritol.

Q. I want the record to be clear that he wasn't working on the propylene oxide triol or tetrol material. A. He did not work on that; that is correct.

Q. When was the next time that someone ran the propylene (315) oxide triol or tetrol urethane—strike that—isocyanate reaction in accordance with your invention after Doctor Herbst did it in April of 1949? A. Nobody did it in my laboratory.

Q. Did anybody with whom you had any connection run it? I am talking about prior to your filing your patent application. A. That is a difficult question for me to decide. I had a connection with Jim Saunders, I was a teacher of his at one time, and he may have run some of these reactions, so I think you need to tell me what you mean by anybody with whom I have had a connection.

Q. I am assuming that if you didn't know about it, you wouldn't have had a connection with them, but I will further refine the question to mean did anybody run the reaction prior to filing your patent application under your direction? A. No.

Q. Did anybody run the reaction at General Tire to your knowledge? A. I don't know the answer to that, for sure.

Q. So that the answer to your knowledge is no? A. To my knowledge I don't know, yes.

Q. So that the only reactions of propylene oxide (316) based triols and tetrols with isocyanates that were run were those run by Doctor Herbst? I am talking about the time before your patent application was filed. A. The only ones of which I had knowledge.

Q. That is all you can answer about. A. That is correct, yes.

(318) Q. If you would, look at Plaintiff's Exhibit 26, which is a letter you wrote. A. Yes.

Q. And if you will look at the, I guess it is, fourth

full paragraph that starts: "In view of . . ."

There you are concerned about an interference with a Dupont patent, and I believe it was the Hill patent, and, apparently, you had had some experience with interference, and you state down in the fourth or fifth sentence that:

"I believe we can make a much better case for the slower development of the idea in the University laboratory."

You were concerned, then, that you hadn't been diligent, weren't you, Doctor? A. I was certainly deeply concerned that we had not (319) yet gotten a patent application filed. That is absolutely correct.

(320) By Mr. Sweeney (Cont.):

- Q. But you had an agreement with Notre Dame which provided that you would got part of any royalties that might result from any invention of yours, didn't you? A. Yes, indeed. That was part of the policy of the University.
- Q. And, as a matter of fact, you did royalties as a result of this invention, and the patent ultimately issued? A. Yes, sir.
- Q. How many dollars in royalties did you get, Doctor? A. I can't recall that precisely. It was on the order—the reason I can't recall precisely is all the patent royalties came in quite some time ago.
- Q. I don't want an absolute figure. A. I have had no royalties since about 1965 because the procedure was or the business arrangement was that companies that got a license would soon turn out to have a paid-up license,

so all this money came in in about the year 1962, when the interference was filed, in 1964 or '65.

As a ballpark statement, I would say it was between fifty and seventy-five thousand dollars.

Q. Which was your share? A. Just my share.

- (321) Q. And you got a share of what Notre Dame got; is that correct? A. A small share of what Notre Dame got; yes, sir.
- Q. So that quite a bit of money was paid as a result of your patents, wasn't it? A. Yes.

Q. By the entire urethane industry? A. Yes.

- Q. Did you know how successful General Tire was in licensing the urethane industry under your patent? A. Well, I have some knowledge of that, of course, because of the way—the terms of the agreement meant that I did have to know something about how much General Tire got, and my understanding is that it is somewhere between five and ten of the producers of the polyol, that is, the polyglycol type product. Somewhere between five and ten of them took licenses under this patent.
- Q. When we took your deposition we talked about the examples in your patent and, if I could find it, I will ask you about those.

Mr. Sweeney: A copy of the patent is marked as Defendant's Exhibit A, if I may have it.

Before I forget, I would like to offer Defendant's Exhibit B in evidence. It is the letter from Doctor Price (322) to Mr. Te Grotenhuis.

Mr. Kelton: No objection, your Honor.

The Court: It will be received.

(Defendant's Exhibit B received in evidence.)

Q. If you will, Doctor Price, look at column 4 in your patent, which contains most of the examples, and then column 5 has one other. A. Yes.

Q. I think you testified in your deposition that Examples 1, 2, 4 and 5 came from work that was done at General Tire. A. May I correct that, Mr. Sweeney?

Q. If I am wrong, please do. A. Examples 1, 4 and 5

were done at General Tire.

- Q. Fine. A. 2 and 3 were examples as the result of our work at Notre Dame.
- Q. Examples 2 and 3 came from the Herbst work? A. That is correct.
- Q. Example 2 is a polyester and that is by the polyester procedure? A. That is correct.
- Q. Then Example 3 is the only example that is of the propylene oxide isocyanate material? (323) A. I have to correct that. It is the only one that we did that was an isocyanate. Examples 1, 4 and 5 were also isocyanate polymers.
- Q. Except that Example 1 is worked on a diol; isn't it? A. Yes.
 - Q. But it is a polypropylene oxide? A. Yes.
- Q. I am talking now about the triol and tetrol materials that Mr. Kelton has been referring to. Example 3 is the only one that shows the use of a propylene oxide triol or tetrol with a diisocyanate? A. That is correct; absolutely.
- Q. That example, then, came from Doctor Herbst's work at Notre Dame? A. That is right.
- Q. In this example it says that fifteen grams of TDI was used? A. Yes.
- Q. I didn't find anything about how many grams of TDI Doctor Herbst used in any of his reports. Where does that fifteen gram amount come from? A. I don't know the answer to that question. You are correct, it was not in his reports. I assume it came (324) from his notebooks or from his experiments in which he made the larger amount. That would be approximately the correct amount to be the size of a tennis hall, 125 milli-

meters, which is the way he made the larger sample, so that I have to admit his prescription that was in his notes for the larger sized sample that was submitted to General Tire, but it was not recorded in the only records that we left, in the summary progress reports.

Q. Did you have Doctor Herbst's notebooks in 1953 when your patent application was prepared? A. I don't

remember that.

Q. Do you know whether— A, I never remember having them in my possession for certain.

Q. Did anybody at General Tire have them? A. Not in my knowledge. We would have given our right arm to have had them.

Q. I am including Te Grotenhuis in the General Tire group? A. We would have loved to have located those

notebooks, so they were not at General Tire.

Q. I am puzzled then, I think you said you supposed that the fifteen gram amount came from Doctor Herbst's notebooks. Now, that would have been getting it out of his (325) notebooks and putting it in your patent application in 1953. How could that have been done? A. By the fact that I might have remembered at that stage. This was still fairly fresh in my memory and I had indeed seen the notebook when I visited his laboratory, so that is why I say I do not know.

Clearly, the answer to your question is I don't know.

I am now only speculating.

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(326) Mr. Sweeney: I will ask the clerk to mark this as Defendant's Exhibit C.

(Defendant's Exhibit C marked for identification.)

Q. Dr. Price, I hand you a print of an article entitled "How Chemists Create a New Product" by Charles C. Price. A. Yes, sir.

Q. Task you if you wrote it? A. Of course.

Mr. Sweeney: I will offer Defendant's Exhibit C in evidence.

Mr. Kelton: No objection, your Honor.

The Court: Received.

(Defendant's Exhibit C received in evidence.)

Q. If you would, please, look at page 133 and in particular at the last full paragraph. Perhaps you would read it, Dr. It might save us a little time. A. The paragraph starting: "Two other . . ."?

Q. Yes. A. "Two other important patent developments relevant to the development of polypropylene oxide poly-

urethane rubber were:

An American patent issued August 9, 1960 to Windemuth, Snell and Bayer, filed in Germany 10 May 1951, and assigned (327) by Mobay and to Bayer. This patent claims products and processes of condensation of diisocyanates with polyalkylene ethers with at least two hydroxyl groups per molecule and of molecular weight above 500. While the claims cover all polyalkylene oxides, the disclosure and examples mention only polyethylene oxide. These German chemists were working in a laboratory which led the world in developing polyester polyure-thanes.

"In a patent filed over two years after our conception and reduction to practice, they had clearly failed to recognize the significant advantages of the propylene oxide unit over the ethylene oxide unit in decreasing water sensitivity and crystallization."

- Q. Thank you. That was your opinion in 1961? A. Yes.
- Q. Is it still your opinion? A. Yes, it is still my opinion.
- Q. I understand from this that as far as you are concerned, the Windemuth patent doesn't disclose the use of propylene oxide? A. No, indeed that is not my

opinion. The Windemuth patent very clearly discloses propylene oxide in the use of the term polyalkylene oxide.

Q. But it doesn't disclose the importance of the (328) propylene oxide? A. I get confused with the legal significance of these terms to I would like to rephrase the thing so I make sure it says what I mean it to say and not what the lawyers mean it to say.

May I do it that way?

Q. Yes. A. Because there is a legal connotation to "disclose" that I think is important here and I don't want to get confused in my poor professor's mind with that legal connotation.

Q. I don't think you are a poor professor at all, Dr.

The Court: I think you are doing quite well, Dr. The Witness: Thank you.

A. To rephrase what I wanted to try to put across here as the idea was that the German patent clearly covered all alkylene oxides as a claim and alkylene oxides include propylene oxide.

What they failed to recognize, fairly obviously from the fact that they did specifically experiment only on ethylene oxides, were the major advantages of propylene oxides as the building block of a network structure. I have indicated (329) what those are in decreased water sensitivity because propylene oxide units have much less affinity for water than ethylene oxide units, because of the methyl group sticking out they don't crystallize as readily and, therefore, they form more flexible chains.

So, I do want to point out and I still believe very strongly that the statement here is correct: That they do disclose propylene oxide by the use of the term alkylene oxide, but they did not recognize the advantages of propylene oxide.

Does that answer your question satisfactorily?

Q. Yes, I think so.

You and I are having the same difficulty then that I had with Dr. Mark. He more or less, also, took the position that Windemuth disclosed propylene oxide because it said alkylene oxide and I gather that is what you have just said? A. Yes, I certainly believe that; yes.

Q. But will you agree with me that there is never the statement propylene oxide in the Windemuth patent? A. The word propylene oxide never appears in his patent;

that is absolutely correct.

Q. When did you first recognize the importance of the water resistant properties of the propylene oxide (330) urethanes? I am talking about the triols and tetrols now. A. I recognized that, I guess, about January 30, 1949, when the basic idea was available because it was known to me that polyethylene oxide was a low—it was known to me that low molecular weight polyethylene oxide was water soluble, whereas a low molecular weight polypropylene oxide was water insoluble.

So, part of the original idea involved the concept that the propylene oxide chain would be a desireable rubber because it would not only be flexible, but was water resistant, which was, of course, very important for prac-

tical applications as rubber.

Q. What I was talking about is when you actually tested the material, but I guess—for that purpose—but I suppose nothing is said about testing for water receptivity in the Herbst reports as no such tests of that kind were done? A. I can't speak for Dr. Herbst, whether he ever actually did an experiment on water solubility. For myself I could say I was so convinced it was resistant to water because the smaller molecules were that I didn't specifically request him to run a water solubility test.

Q. You mentioned a term a few minutes ago, the methyl groups in the propylene oxide material. I wonder (331) if you would explain a little more fully the importance

of the methyl groups and these are the methyl groups that are sticking out from the chain as the result of the propylene oxide? A. Yes, every propylene unit has two carbons that are in the backbone of the chain and a methyl group that sticks out in the chain.

Q. Right. A. And the importance in my original idea, in my concept of this whole invention, the importance of the methyl groups was due to the fact that that induced a lack of symmetry in the chains which made it very difficult for them to fit into a crystal structure.

It also introduces a greater degree of resistance to water because the CH3 group, the hydrocarbon, is known to be the kind of a group which is resistant to water. So that the idea that the methyl groups would do what I said they did in this article was not an unreasonable idea.

Q. But the presence of the methyl group, then, is important for making urethane materials? A. It is important for making a polyurethane material that you want to be resistant to water.

(333) By Mr. Sweeney (Cont'd):

Q. The Hill patent, of course, was cited against your application in the Patent office? A. The Hill patent covers the preparation of (333-a) disocyanate extensions of diols and I think this was indeed cited against us because some of our original claims covered that material.

Q. But insofar as the claims that you ultimately got, you argued over Hill? A. Oh, yes.

(334) Q. Do you recall what your argument was? A. I think the main argument was that we were using triols and tetrols.

By Mr. Sweeney:

- Q. Do you think it would be obvious to do what you did from a combination of the Sokol and Hill patents, insofar as your propylene oxide triol tetrol isocyanate work was concerned?
- (336) A. The answer to your question has to be no in 1949 because the Hill patent didn't issue until 1955 and the Sokol patent until 1950, so obviously they could not have been known to me in 1949.
- Q. Let's take 1953, when you filed your patent application, then. A. I suppose by the time these patents issued, it would become a rather interesting and fairly obvious thing to try to put the two together to make a network structure polyurethane after they had both issued.

The Court: Is the answer to that, to make sure I understand it, as I understood the question it was putting the two together, the Hill and the Sokol patent together, would that have been obvious to you? Was that the question?

Mr. Sweeney: That is what I asked him, yes.

The Court: Your answer is that it would have been? The Witness: Certainly, in the light of the teaching of Bayer that you could make a network. Hill teaches that you are making a polyurethane from a polypropylene oxide and Sokol teaches you how to make a branch chain (337) polypropylene oxide, so the three elements together would constitute what was my invention in 1949.

(345) Mr. Kelton: As I understand it, Jefferson Chemical is jointly owned by American Cyanamid Company and by Texaco and I assume that it's through holdings of stock.

Is that correct, Mr. Sweeney or Mr. Ries?
Mr. Sweeney: I don't know the answer. Maybe
Mr. Ries does.

Mr. Ries: The practice at Jefferson is to have an executive committee composed of the president of the company and two members of the board of directors, one of the members being an officer of Texaco and the other being an officer of American Cyanamid.

The executive committee considers matters relating to Jefferson. These men are also, the executive committee, are also on Jefferson's board of directors.

Mr. Kelton: Well, my inquiry went to whether or not Texaco is a major stockholder in Jefferson and whether American Cyanamid is also.

Mr. Ries: Yes. Each own 50 percent of the stock.

(349) Continued Cross Examination by Mr. Sweeney:

Q. I want to show to you part of the file history of your application which deals with an affidavit under Rule 31.

Mr. Sweeney: This is what you call an affidavit, your Honor, that you use when you are trying to swear back a reference.

The Court: All right.

Q. I have opened it to that page, just to make it easier, Dr. Price.

The Court: What page is that?

Mr. Sweeney: Page 28 of the file history, your Honor.

- Q. Dr. Price, is that an affidavit that you signed? A. Yes, sir.
- Q. On or about the date which I can't read in 1956? Maybe it's clearer on your copy. A. The 11th of July, I believe. It's the 11th of July, 1956.
- Q. Attached to that affidavit are a group of exhibits. A. Yes.
- Q. The first one, which is marked Exhibit A, and that would be an exhibit to your affidavit, I take it—(350) A. Yes.
- Q.—is a copy of a progress report from you and Dr. Herbst, am I correct? A. That's right.
- Q. If you would, look at Exhibit 21, Plaintiff's Exhibit 21, and tell me, is that the same progress report as Exhibit A to your affidavit? A. Yes, it is.
- Q. If you would, turn to Exhibit B and look at Plaintiff's Exhibit 19 and tell me if Plaintiff's Exhibit 19 and Exhibit B are the same. A. They are evidently identical except that the date has been cut out of Exhibit B and there have been some vertical rules put in the table. Otherwise, I believe it is identically the same document.
- Q. Then, if you would, look at Exhibit C, which is the letter which you wrote to Mr. Te Grotenhuis which we marked this morning as Defendant's Exhibit B, which I believe you still have.

You remember that this is a type off because we couldn't read the letter that was in the files. A. Oh, yes.

Mr. Kelton: We will concede they are the same papers, Mr. Sweeney, except that Exhibit B does not bear a (351) "received, Division 60" stamp and the date which—

The Court: I can't hear you. I'm sorry. I heard that you concede.

Mr. Kelton: We will concede that this Exhibit C is the same letter as Defendant's Exhibit B except that Exhibit C does not have "received August 15, 1956, Division 60" stamp and in place of that it has a date, Defendant's Exhibit B has a date which is covered on this, as is customary when submitting documents to the patent office for this purpose.

Q. And then, Dr. Price, look at your affidavit, Exhibit D, and tell me if it is the same as Plaintiff's Exhibit 23.

Mr. Kelton: We will concede that it is except that it does not have the same handwriting on page 2 and the date of August 8, 1949, on page 3 of Plaintiff's Exhibit 43 has been covered on Exhibit D.

The Court: Well, I think that the purpose of this is to establish your right to cross examine Dr. Price and be within the limits—

Mr. Sweeney: I was being careful, your Honor.

- Q. Dr. Price, the final report, the summary report that you submitted, which was Plaintiff's Exhibit 24, was not part of your affidavit in the patent office. (352) A. That's correct.
- Q. Do you know why it wasn't? A. No. I do not know.
- Q. Who prepared the affidavit? A. I am almost certain it was Mr. TeGrotenhuis.
- Q. Did he have Exhibit 24 at the time he prepared this affidavit? A. I cannot be certain of that. These were sent, as I said, to Mr. Swart. I assume he had access to them.
- Q. What was the outcome of your attempting to swear back of the Hill reference, the Hill patent, which is,

Dr. Charles C. Price, for Plaintiff, Re-direct

here, Plaintiff's Exhibit 9? A. I can only tell you what I understand that to be.

Q. Please do. A. I understand it to be that we were unsuccessful in being able to establish our priority to the linear diglycol materials which were included in our patent application; the linear diol materials.

(355) By Mr. Sweeney:

Q. Did you argue that your invention was patentable over the Hill patent? A. Certainly.

(357) By Mr. Sweeney:

Q. I am not sure that you testified about this. You may have already testified, Dr. Price, but are you still a (358) consultant for General Tire? A. Yes.

(360) Re-direct Examination by Mr. Kelton:

(361) Q. Dr. Price, I don't recall whether I have asked you whether you have any familiarity with the Heiss patent, Plaintiff's Exhibit 1. Do you? A. Yes.

Q. With regard to the singling out of propylene oxide, can you tell me whether that is done in your patent as compared with ethylene oxide? Your patent being Defendant's Exhibit A. A. In my patent there is no example involving polyethylene oxide and no claim to polyethlyene oxide.

Q. Could you make any comparable comment with respect to the Heiss patent, Plaintiff's Exhibit 1? A. In many of the examples of the Heiss patent, they use either propylene oxide or ethylene oxide more or (362) less interchangeably as the backbone of the polymer structure.

(363) By Mr. Kelton:

Q. During cross examination Mr. Sweeney showed you a paper which has been admitted as Defendant's Exhibit E.

Did you receive that letter shortly after the date it bears? A. Yes.

Q. Do you recall whether you agreed or disagreed with Mr. TeGrotenhuis? A. There was certainly one part of this letter with which I disagreed—the sentence in which he said the work done at Notre Dame would never win the interference for a junior party.

(364) It seemed to me that the facts were pretty clear. We had the idea. We had disclosed it in documents. We had made a rubber but whether he was arguing there on the basis of some kind of law, I don't know but, as far as facts were concerned, I argued with him that we ought to be able to prove that we had done this, that we had made a rubber in 1949.

I know we had an argument about this, a discussion about it maybe is a more polite way of describing it.

Re-cross Examination by Mr. Sweeney:

(367) By Mr. Sweeney (cont'd):

Q. Did you write a letter, Dr. Price, in response to Mr. TeGrotenjuis's letter which is marked Defendant's Exhibit E? A. I don't remember. May well have.

Q. You don't have it if you did? A. I don't have it with me.

Q. You produced all of your documents— A. I produced all the documents. If it isn't among the documents, I did not write a letter. I answered that by the discussion with him at General Tire.

(369) RALPH L. HERBST, called as a witness by the Plaintiff, being first duly sworn, testified as follows:

Direct Examination by Mr. Kelton:

- Q. Will you please state your name and residence? A. Ralph L. Herbst, Jr., 34 Harmon Avenue, Pelham, New York.
- Q. What is your present position? A. Manager of new polymer development, Hooker Chemical Corporation, Stamford, Connecticut.
- (370) Q. Would you state your education? A. Bachelors degree in Chemistry, Muhlenburg College, Allentown, Pennsylvania, October 1943.

Phd, Organic Chemistry, Notre Dame; Notre Dame, Indiana, in June of 1949.

Also, Alexander Hamilton Institute, New York, 1957.

- Q. You say you got your PhD in organic chemistry at Notre Dame in June of 1949. Do you recall when you completed your work and qualification for that degree? A. My requirements were completed at the end of February. It included the completion of my thesis on the kinetics of peroxide decomposition and I had to have this completed, everything bound and in the Dean's office by March 1.
- Q. When you say the end of February, what year are you referring to? A. 1949.
- Q. From the end of February, upon your completion of your thesis requirements, did you thereafter undertake any work of any kind? A. Yes, sir. Under Professor Price's direction and supervision, I undertook work on the polymerization of propylene oxide in the presence of pentaerythritol and other (371) polyhydric alcohols with subsequent reaction of that product with diisocyanates, monoisocyanates and also dibasic acids and anhydrides.
- Q. The dibasic acid would give you what is called an ester— A. A polyester.

Q. A polyester linkage, is that correct? A. Yes.

Q. And the diisocyanates would give you what kind—A. The polyurethane.

Q. Do you remember about when your first engagement in this project was? A. Yes. I started about March 1 and carried out the program up until the last week in August, just before Labor Day in 1949.

Q. Do you have any special reason to recall that you carried it up to about the last of August of 1949? A. Yes, I do. I had accepted a teaching position at Utica College, Utica, New York, and I was anxious to finish my work and start my teaching assignment.

Q. Did you go to Utica College? A. Yes, sir.

Q. About when did you go? A. It was right after Labor Day.

(372) Q. Can you tell me, as you recollect, how your connection with this work or project began? A. I had just deposited my thesis in the Dean's office and met with Professor Price to discuss another research project that he had n mind. He told me he had a real good practical idea of how to make a new rubber and then he went on to explain his concept was to take propylene oxide, a volatile liquid, react it with sodium metal or other catalyst, such as sulphuric acid.

The purpose of this was to make a condensate with hydroxyl groups. He was concerned about achieving a high enough molecular weight so good rubberry characteristics could be obtained.

We talked about various catalyst systems that we felt could be tried in the hope of achieving this high molecular weight. He reasoned that with—I came back to him somewhat skeptical about this concept because at that time all of the rubbery materials, elastomers, were or contained only carbon and hydrogen and contained a conjugated system of either isoprene or 1,3 butadiene.

His concept seemed at that time like a real revolutionary idea. So, I was somewhat skeptical and he said, "Well, if we can't get a high enough molecular weight; that is, make a giant molecule here," that we would react the (373) hydroxyl terminals with disocyanates or also possibly with a dibasic acid, acid chlorides or anhydrides to increase the molecular weight into a giant molecule so we could obtain a rubbery characteristic.

That was his original concept at that time and then I went down to my basement laboratory and procured the equipment and chemicals which I needed and I do recall rather vividly some of the mechanical difficulties I had in working with the volatile propylene oxide.

My initial experiments were with—were carried out in glass equipment, three-necked flasks, approximately 125 up to 300 milliliters equipped with an agitator, a reflux condenser and a thermometer and I carefully cleaned the sodium metal and dropped it into this reaction mass and stirred it gentle when I noticed it become a milky white, more turbid, generated heat initially and then I had to keep on stirring it for a longer period of time to get a complete reaction.

In another series of experiments, I ran—I borrowed a stainless steel pressure bomb from Dr. Kenneth Campbell. His office was up on the second floor in the old Notre Dame Laboratory building, and I borrowed his pressure bomb and in this case, all of the reactants were put into bomb and the bomb sealed tightly with wrenches, the bomb (374) immersed in an oil bath and the oil bath heated over a period of time and I recorded the pressure and the temperature conditions and then also used that reaction product as a starting material to react with the diisocyanates and also with the anhydrides and dibasic acids.

During this period I received some needling and some barbs by fellow graduate students and also by other professors who insisted that this just—this wouldn't work.

Some went to the extreme of calling me a stupid ass, so I continued my effort and I was delighted that after about one month, I had been able to make, in small quantities, some rubber balls which I took up to Dr. Price and bounced them off the top of his desk and he became quite enthusiastic and directed me to make a larger quantity. He said a ball of the stuff.

He directed me to determine the molecular weight of the propylene oxide condensate, the molecular weight of the condensate. By that I mean the reaction product of the propylene oxide with the pentaerythritol. And also he directed me to see, to try to determine how broad a scope this reaction had and that's when he specifically told me to do the work on—with the anhydrides and polyesters.

Q. Now. Dr. Herbst, you referred to these rubber balls

that you bounced on Dr. Price's desk.

(375) How are those made, to the extent that you recall? Can you tell us just what you did? A. Yes. My initial experiments were run on a test tube scale and in a small flask scale where I took the propylene oxide pentaerythritol condensate and placed it in the bottom of a long test tube and then with an eyedropper added the diisocyanate to that flask, to that test tube.

I found that the reaction generated a lot of heat. I was holding it in my hands and I could feel the heat generated and I swirled the reaction mass and it became more and more viscous and I had to cool it under the running tap, tap water in the laboratory, and by cooling it under tap water and adding the diisocyanate with an eyedropper, I then could get control of the reaction, handle it, and then make—Dr. Price did emphasize to me

to make sure that I had a complete reaction of the diisocyanate so I could continue to add the diisocyanate until it formed into one solid mass, making sure that an excess amount of diisocyanate was added to make sure I had a complete reaction.

The next step was, after this solid mass had formed, I simply took the flask, cooled it and smashed it to break the glass. I picked the glass off the outside and I had my little rubber balls.

To make the larger quantities that Dr. Price had (376) requested, those experiments were carried out in a larger flask, about 100 to 125 milliliters and, instead of using an eyedropper, the diisocyanate was added through a burette until I was sure that I had added an excess of the diisocyanate to make sure that I had a complete reaction.

Again, I had to use external cooling water to keep the reaction under control.

I also had to—I found it very important to add a solvent, either alphachloronaphthalene or diethylphthalate. The reason I did this was to prevent the heat generation during the initial stage of the reaction and this gave me better control.

At the completion of the reaction I simply took the reaction mass in my flask and I slammed it on the laboratory bench top to break it, peeled off the outside glass and I had my larger balls which Dr. Price had requested.

I found it very important to keep my equipment dry. I went to great pains to flame-dry the equipment. If I did not keep it dry, I would get foamy gels, foamy products.

Q. Now, you have referred to the use of the solvent in the flask or larger scale operations. A. Yes.

Q. Let me ask you, did you use a similar solvent in (377) order to obtain your bouncing balls in the test tube scale? A. My recollection is that when I used the

solvent, alphachloronaphthalene, or diethylphthalate, I got the bounding rubber ball. When I used the acetone, I did not get a bouncing ball. I do recall that with the alphachloronaphthalene I had a darker colored product, whereas with the diethylphthalate it was lighter in color.

Q. Did you make any attempt to remove the solvents from any of the balls, small or large? A. Yes, sir, I did. For both the small balls and for the larger balls, I hooked up the reaction vessel at the end of the reaction and attached it to a vacuum line that I had hooked up in the hood in my laboratory.

This was hooked up with a vacuum pump that pulled the vacuum in a range of half a millimeter or so—between a half and one millimeter, in that range—and I would keep the reaction product in there for a period of four or five days.

I found that if I did not do that, why the outer surface of the rubber ball would have a tackiness, a tacky feel.

Q. Do you recall whether or not you recorded your work in the period of March through August, 1949, in a notebook? A. Yes, sir, I did. I used a standard notebook as (378) issued by the stockroom at Notre Dame. It was a bound notebook, spotted black and white, with a number and with numbered pages.

All of my experiments were carefully recorded in that notebook. This was a different notebook from the one which I had used for my thesis work on the Kinetics of Peroxide Decomposition.

Q. Let me get back to your treatment of the balls under a vacuum.

I think you indicated that tackiness would disappear after it had been treated. Let me ask you, what did the disappearance of tackiness indicate to you, if anything? A. It indicated to me that there was some—some of the solvent had probably adhered to the surface of the

rubber ball and I used this vacuum treatment simply to remove as much as I could of any residual solvent. This was a slow process.

Q. Did it extend over hours, days? A. Four or five days.

Mr. Kelton: I am about to get into another area, your Honor. It's practically that time.

The Court: We'll take a short recess, ten minutes.

(Recess.)

(379) By Mr. Kelton:

Q. Now, Dr. Herbst, I refer to your testimony which I take it to mean that there was a notebook, a separate notebook in which you recorded your operations between about the beginning of March 1949 through August, 1949.

Can you tell me-

The Court: You nodded your head. Was the answer "Yes"?

Mr. Kelton: I didn't get your Honor.

The Court: You asked a question and he nodded his head. There was no indication in the record as to nodding his head.

A. Yes, I did have a separate notebook.

Q. Dr. Herbst, let me ask you when, to the best of your recollection, did you last see that book, and tell me the occasion, if you can't tell me the time. A. I last saw the book during the last week in August before I and my family packed up and left Notre Dame to go to Utica, New York. At that time I took all of my notebooks, my notebook for this work on the propylene oxide

polyurethane work and also all of my notebooks from my thesis work, and also my last report, and placed all of this in Dr. Price's outer office. At that time he had an inner office and his secretary had an outer office, and I simply (380) stacked all these notebooks and my last report and samples on a pile, and I do recall that Dr. Price was not present at the time.

Q. Did Dr. Price ever ask you if you had the notebook? A. Yes, he did.

- Q. Did you or didn't you? A. No. I have not seen the notebook since. He asked me, oh, it was around 1960 or '62, he called me on the telephone. I was living over in Huntington Valley, Pennsylvania near Willow Grove, and I told him my recollection was that the last I had seen them, I had put them in his outer office and he said, "Well, to be absolutely sure, would you and your wife please make a house search?" and we did, but we didn't find any notebooks. We had moved from Utica—had several moves in between this period, and my wife has no recollection of seeing the notebooks either since we left Notre Dame.
- Q. Do you have any recollection of seeing— A. No, I don't. No, no recollection.
- Q. Can you tell me what your relationship was with Dr. Price or his to you in this particular work? A. Well, Dr. Price, of course, was my professor for my thesis work and teacher, and he was my direct supervisor (381) of this program. He supplied me with some of the chemicals which I used. He supplied me with the disocyanates, the TDI and the MDI. They were experimental materials that he had obtained with the du Pont labels on the bottles.

We had discussions in his office, very informal type discussions in his office, in the hallway, down in my laboratory, and when he wanted me to perform some experiment with him, we had a note system that we used.

He would come down to my laboratory and say, "Ralph, here are the samples of what happened in this experiment," of what happened and so forth, and this was the system that we used to communicate because in carrying out these experiments, this was not a 9 to 5 joh; when I started work in the morning, I would like to keep running all the way through until I had completed that particular series of experiments and many times this took a runover into the evening and late at night to finish the experiment.

Q. Can you tell me whether or not you were paid for the work you did during this period? A. Yes. That I remember very well, sir. \$250 per month, which was an increase of about \$25 per month after I had completed my requirements for my doctorate. I have received no financial—no money since this work was completed at the end of August, 1949.

(382) Q. Now, there have been questions raised here as to the matters of water solubility, thirst for water and that type of thing.

Did you ever make any tests of any of the products that you made which would help us at all in determining that matter? A. Yes, sir. I certainly took the both products formed by the reaction of the propylene oxide pentaerythritol with diisocyanate, a polyurethane product, and I also—

Q. With solvent or without solvent? A. With solvent and without solvent. And they were insoluble in water. In fact, I checked a number of different common solvents in the laboratory and did not observe any solubility, not only in water but in other common solvents.

Q. Did you report others than entries in your notebook with regard to this work? A. I submitted a series of progress reports to Professor Price during the course of this work. Those progress reports were based upon the experiments which I had performed and which I had recorded in my notebook.

The progress reports were rather terse, concise reports. The reason for that was I had written a wordy. voluminous thesis, over three hundred pages, and Dr. Price was rather critical and asked me to keep the wordage down.

(383) Q. How were these progress reports submitted by you, in what form were they? A. They were submitted in 8 by 10 paper, which I typed myself. I am not a very good typist, but the hunt and peck system, which I typed at Notre Dame, and I resided with my family at that time in Apartment 7-C—

The Court: Mr. Kelton, there is no dispute as far as I know that the exhibits that you introduced were in the progress reports that were made by Dr. Herbst. I find this cumulative and needless since you have established it, it seems to me, by Dr. Price.

By Mr. Kelton:

mirate to take the terms of the

Q. Now, Dr. Herbst, I show you Plaintiff's Exhibits 18, 20, 22 and 24, and ask you what they are. A. These are copies of the progress reports on the propylene oxide pentaerythritol polymer and my experimental work of reacting that condensate with the isocyanates and the dibasic acide and anhydrides during this period from April to August in 1949. Some of the writing on these reports is not mine. It looks like it has been edited by Professor Price but the signature at the bottom of each report is my signature.

Q. Can you tell me whether or not the typing is your typing on these? A. Yes, sir, it is. It is my typing. (384) Q. Can you identify in any of these reports the bouncing rubber hall material if you reported on it here? A. Yes, the bouncing rubbery halls was made with the experiments in the presence of the solvents, alphachloronaphthalene and diethylphthalate as recorded on the bottom

of the page of this Exhibit 18. That records my early -my first series of experiments.

Q. Can you tell me whether or not you had occasion to make the same material in larger amount? A. Yes, These experiments were made in the test tube that I had described earlier when Dr. Price asked me to make this larger quantity, a ball of the stuff. I repeated this work in a larger scale, in a hundred milliliter, 125 milliliter flask, a larger quantity.

Q. I don't find any report in the succeeding progress reports of your having made that larger quantity. Can you tell me why that is? A. Because I had used exactly the same procedure that I had used earlier here in this first report, number 18. I didn't feel it was necessary to report the larger quantity. There was no new information that I had gained other than I had made it on a larger scale, so that I could give a larger quantity

Q. Now, on Exhibit 20-B, Doctor, I call your attention (385) to the statement on page 2 under the heading "Reaction Between Propylene Oxide Polymers and Diisocyanate Compounds." You have reference there to the hard brittle polymers using a large excess of propylene oxide as compared with pentaerythritol. Can you tell me what that had to do with? A. We did not know the ratio of propylene oxide to pentaerythritol that should he used. This was why we carried out experiments with different amounts of pentaerythritol present.

When the reaction was carried out in the absence of solvent, I obtained the hard, brittle materials, and this

refers to those experiments.

Q. I notice in Plaintiff's Exhibit 18, in one place you have in typing, in several places you hard, br. What did you use br to designate? A. Brown.

Q. I notice over here in the lower portion on the right hand side you used the letters bl. What did you mean

there? A. Black.

to Professor Price.

Q. I notice on Plaintiff's Exhibit 22 there is a report of molecular weight determination in preparation of deionized propylene oxide pentaerythritol polymers, things such as that; were they done in between the period of the preceding report (386) or-and August 6, 1949-That was done between the May and August Professor Price had asked me to determine the molecular weight of the condensate of propylene oxide with pentaerythritol. To do this we felt it was important to purify this material and this is why I used this technique of dissolving the condensate in dioxide as a solvent, passing it through a Rohm and Hass ion exchange resin to remove the base which was present. I did this to make sure I could get a more accurate determination of the molecular weight. I also did this for another reason. Professor Price had requested me to prepare the polyester polymers and to do this I had to first remove any residual base from the propylene oxide pentaerythritol condensate to make sure I got a good reaction between the condensate and the dibasic acids and anhydrides.

Q. You have referred to the work on reacting the propylene oxide pentaerythritol condensate with anhydrides. A. Yes.

Q. Let me ask you: would that give you what is called a polyester? A. Yes, sir. It would.

Q. Did you do the polyester work in the same equipment, namely, test tubes and flask, that you did the polyurethane (387) work? A. The starting material, that is, the condensate of the propylene oxide and the pentaery-thritol was made in the same equipment. However, for the reaction with the anhydrides, to make the polyester, those were made in open Petri dishes because in that case what I had to do was to intimately mix the two reactants with stirring and agitation, stirring rod, and let the mass simmer for a fairly long period of time to drive off water which was the other reaction product,

and as I continued to stir with the stirring rod, the mass, again, would solidify into a rubbery product in an open Petri dish.

One other difference was instead of the—in this case I had to apply heat, much more heat than I—than with the polyurethanes to get this reaction to completion and to drive off the last amount of water from the reaction mass.

Q. Now, can you tell me if you recall the comparative physical properties as you observed them of the rubber hall material, I will call it, which is reported as made at the bottom of Plaintiff's Exhibit 18, which is the April 19, 1949 progress report? Those on the one hand and polyesters on the other. A. To my best recollection, the rubbery products made by the polyester reaction were a softer, pliable type of (388) rubber; they did not ball up. I tried and I could not make a ball from this material, whereas in centrast to that behavior the polyurethanes produced two different pro-Those made in the absence of solvent or in acetone were hard materials, insoluble in water and most common solvents. In the presence of the alphachloronaphthalene or the diethylphthalate as solvent, there I obtained also rubbery materials. As I recall, they were somewhat harder than the ones with polyesters, but they had one additional characteristic, that was they were bounceable. This was not a big bounce like a golf ball but they were-you could bounce them on the table top. This was the main distinction between the rubbery product obtained from polyesters as compared with those products obtained from the reaction with diisocyanates with one exception to that statement: In the case where I used a monoisocyanate, I got a waxy material.

Q. Now, Doctor Herbst, in the case of Exhibits 18, 20, 22 and 24, Plaintiff's Exhibits, in each case do you find that you signed it? Your signature appears? A. Yes. Yes.

Yes, my signature is at the bottom of each report. Q. When did you sign these reports? I mean in re-

lation to when they were submitted. A. I signed them

right after I finished the typing.

(389) Q. I notice that Exhibit 24 is headed "Summary Report." Can you tell me whether or not you intended that report to cover and summarize all of the work 'you had done? A. No. That report was not intended to summarize all of the work.

Dr. Price had asked me to report to him the results of experiments that I had not reported earlier. He also asked me to give him a description of the chemicals and reagents which were used and altogether more detail with respect to the manipulation and the process conditions that were used. He knew that I had been carrying out this work, some of it being in various stages of completion, and he just wanted me to report to him those items that I had not reported earlier. In that respect this was more like the last or a wrap up report and he also asked me to be terse and brief because all the detail was in my notebook.

(390) Cross Examination by Mr. Sweeney:

Q. Do you still have the reports in front of you, Mr. Herbst? A. No, I do not.

Q. It would be easier if you had them.

The Court: 18 to 22.

Mr. Kelton: I hadn't given Dr. Herbst the retypings that Dr. Price made, you see. They may be included; I don't know. He can pick out his own.

Mr. Sweeney: I am talking about Plaintiff's Exhibits 18, 20, 22 and 24.

A. 18, 20—

The Court: 22 and 24. The Witness: 22 and 24.

Mr. Sweeney: If you have any more, you have too much.

- Q. You considered the rubbery materials that you got as a result of the reaction shown on Plaintiff's 18 to be the best material that you made, didn't you? I am talking (391) about the two that are listed at the bottom of the report, those made in alphachloronaphthalene and diethlphthalate. A. Yes, I did, because these were the ones which bounced.
- Q. You also testified that you put your disocyanate into these reactions by an eye-dropper. A. In my initial experiments.
- Q. Yes. Did you use an excess of diisocyanate? A. Yes.
- Q. Why didn't you put that in your report? A. It is in my notebook.
- Q. That isn't the answer to my question. A. Because I wasn't requested to put that kind of detail in my progress report. Those details were in my notebook. But I knew full well that I had to have an excess to make sure that I was getting a complete reaction, and I had a fully cured system.
- Q. Well, I guess we could agree with each other, though, that you didn't put the fact that you used an excess in your report? A. I mention it in the case involved with the MDI.
- Q. Does that mean you used an excess of MDI? A. Yes.
- Q. An excess over what? (392) A. An excess over the calculated amount needed to make sure I was getting the complete reaction of the two NCO groups of the MDI or the TDI with the hydroxyl content present in the propylene oxide pentaerythritol condensate.

Q. It's been established, you may have even been here today when Dr. Price talked about the fact that, examples two and three of his patent came out of your work. Did you hear that? A. I heard that today, yes.

Q. I hand you Defendant's Exhibit A, Dr. Herbst,

and I will ask you to look at Example 3.

Did example 3 come from your work? A. I can't answer your question. I didn't write the patent application.

Q. Well, you could look at the example. Did you do what it says in example 3? A. I did this, a very similar experiment in my scale-up experiments which I described earlier, using the 125 milliliter flasks. These were the approximate quantities that would be used in that experiment; that series of experiments.

Q. Since the scale-up experiment is not in any of your reports, I presume, therefore, that the work that you did that resulted in example 3 is not in any of the reports. A. It was written up in detail in my notebook. (393) Q. But it's not in the reports? A. It's not listed

in detail in the reports, no, sir.

Q. Was it listed in any kind—you say it's not listed in detail. A. In these particular progress reports.

Your Exhibits 18, 20, 22 and 24. But, it is written

up in detail in my notebook.

Q. The missing notebook? A. The missing notebook.

Q. I notice that in example 3, fifteen grams of TDI is recited. Is that the amount of TDI that you used? A. It seems like it would be the approximate amount. I don't recall the exact amount, no, but it would—on scale of 125 milliliter flask, assuming about 100 grams of material, it would probably consume somewhere around 10 to 15 grams of diisocyanate to get a complete reaction.

Q. But that amount is not given either in any of your reports— A. It is not given and specified in these par-

ticular exhibits-18, 20, 22 and 24.

Q. Look, now, at Plaintiff's Exhibit 18 again, and I want to call your attention to the top part of the report which is where you are discussing making the condensate with the pentaerythritol and propylene oxide. (394) What form was the pentaerythritol that you used? What form did it take? A. I added it as a solid.

Q. It was a solid? A. It was a solid.

Q. Could you describe it? A. White, a pale yellow solvent, powder.

Q. Powdery, flaky or— A. It was a pale yellow powder, as I recall.

Q. And then if you will look at your report here, you say in the last sentence of that paragraph that "fivetenths gram of unreacted pentaerythritol was recovered from the reaction in Number 1." A. Those reactions were carried on under pressure at a higher temperature.

Q. But what form was the recovered pentaerythritol in? A. It was recovered in a solid form.

Q. How did it compare to the pentaerythritol that you started? A. It looked about the same. And it settled down to the bottom of the pressure bomb and when I observed this was when I poured the contents of the bomb out into a receiver and I noted that this residual solid was at the bottom of the pressure bomb.

(394) Q. Would you expect that any additional pentaery-thritol would have been in solution in the liquid that you had? A. Conceiveably some could be in solution.

Q. Did you do anything to determine whether there was or wasn't? <A. Well, the solution that I took was perfectly clear and when chilling it, I observed no more precipitation so I assumed that it was—had all precipitated out.

When I cooled it down to room temperature and below, I put it under a cold tap.

Q. When you mixed the propylene oxide, which was

a liquid—am I correct? A. Yes.

Q.—with the pentaerythritol, did the pentaerythritol dissolve in the propylene oxide? A. When I carried out the reactions in the three-necked flask, when I added the sodium metal, what—my procedure was this:

I would add the propylene oxide to the reaction vessel with stirring. I then would add the pentaerythritol with continued stirring.

Q. This is the point I am asking you about. A. Then I would add as the last ingredient small pea-sized pieces of sodium metal.

(395) Q. Let's go back to the step just before you added the metal.

What I wanted to know was did the pentaerythritol dissolve in the propylene oxide. A. I don't recall that.. I don't recall. It's been too long. I do know that after I added, the most significant part or observation was when I added the sodium metal, the reaction mass turned from a colorless liquid to a white milky fluid.

Q. I don't think I asked you about that yet, Dr. Herbst,

so will you just hold it.

I think if you will look at Plaintiff's Exhibit 24 on page 2, you say in the section under sodium as catalyst—do you see where that is?

You say that there fluxing commenced at 18 to 20 degrees. What ingredient was boiling at that temperature? A. It must have been the reaction product of the sodium metal and the pentaerythritol in the presence of the propylene oxide. I doubt if it was the propylene oxide, because that boils at about 35 degrees C.

Q. That is correct.

The Court: Excuse me. Mr. Kelton or Mr. Blecker, is it possible for you to get me a clearer copy?

(Discussion off the record.)

- (396) Q. You said then that you think it was a reaction product of the sodium and the pentaerythritol? A. And the propylene oxide. I didn't know which it was. I couldn't tell which.
- Q. Propylene oxide, we have agreed, boils at about 35. A. I said I don't think it was propylene oxide because that boils at about 35 degrees C.
- Q. Now, Dr. Herbst, to try and get this straight, do I understand you think the material that was boiling at 18 to 20 degrees was a reaction product of the sodium, the pentaerythritol and the propylene oxide? A. I couldn't tell which.
- Q. You don't know what it was? A. I couldn't tell. All I know is, I put in the flask propylene oxide, pentaerythritol and sodium metal. The only thing I could be sure of, it was not the propylene oxide, because that boils at about 35 degrees centigrade.
 - Q. The pentaerythritol is a solid? A. Yes.
- Q. And so is the sodium. A. And so is the sodium. It could have been some reaction product of the sodium with the propylene oxide or possibly with the pentaery-thritol. I have no way of knowing.
- Q. Going back to the reaction you described on Plaintiff's (397) Exhibit 18, do you know, do you have any idea why you recovered the pentaerythritol from the reaction listed as Number 1? A. Listed in Number 1?
 - Q. It is listed as Number. A. Why I recovered it?
- Q. Right. A. I was trying to get an idea, trying to measure how complete the reaction was and when I saw this solid residue at the bottom of the pressure bomb, I simply poured it out and dried it and weighed it.

- Q. What I really meant was, do you know why it didn't react? A. No.
- Q. Do I understand, then, that some of the pentaery-thritol never dissolved in the propylene oxide? A. I can't answer your question because I can't look inside a steel bomb under pressure that is heated up to 120 to 140 degrees.

I don't know if it dissolved or not.

- Q. You ran the reaction, though, as you testified a moment ago, in a three-necked flask? A. That was a different series of experiments.
- Q. And you could see whether or not the pentaerythritol (398) was dissolved. A. Well, it was difficult to see because the reaction mixture very quickly became milky and I couldn't tell if it had dissolved or not. It's like trying to see if it dissolves in a milky solution. I couldn't tell if it dissolved or not.

I do recall this: That in the reactions run in the flask, in the glass flask, at atmospheric pressure, in those cases I did not get any residual pentaerythritol after the reaction was completed under the conditions that I mention in here.

These reactions were carried out for a period of time and at the end of that reaction, I did not observe any residual pentaerythritol. I didn't recover any, so I assumed that it reacted.

Q. What reaction are you talking about now! Is it in one of these reports? Can you point out the report! A. The reaction on Exhibit 18 in the first paragraph, the subparagraph 2, a reaction at room temperature for three days, followed by heating at 110 to 115 degrees.

In those series of reactions, I do not recall seeing any residual, unreacted pentaerythritol.

(399) By Mr. Sweeney (con't.)

Q. Look at Plaintiff's Exhibit 20, now, if you will. On page 2 there is a heading "B," and it is using a propylene oxide pentaerythritol, et cetera.

Do you see where I mean? A. Subparagraph B?

Q. Yes. Does this describe additional reaction that you ran with propylene oxide and pentaerythritol, or is it the same reaction that you ran and reported on in Plaintiff's Exhibit 18? (400) A. If I understand your question—are the experiments on page 2, paragraph b in Exhibit 20 the same as I described on Exhibit 18, my best recollection is that this is a continuation of that series but where I am changing the ratio of the propylene oxide to the pentaerythritol—

Q. You report about the same results in paragraph b of Exhibit 20, don't you? A. I report that the product in paragraph b gave a rubbery material in the presence of diethylphthalate but hard, brittle polymers when no

solvent was used.

Q. Which is about the same thing you reported in Exhibit 18? A. It seems to be about the same, yes, the best I can remember it.

Q. Look at the section under, call it, paragraph c. There you are reacting propylene oxide with itself and then with disocyanate, aren't you? A. What do you mean by propylene oxide by itself? I never reacted propylene oxide by itself. I always had present the propylene oxide, the sodium metal or some other catalyst plus pentaerythritol.

I don't recall any reactions where I reacted propylene oxide by itself. If you mean just propylene oxide reacting

with more propylene oxide-

(401) Q. Which it would do? A. I didn't try that. All my experiments were done with propylene oxide, a catalyst such as sodium metal or sulphuric acid or boron trifluoride etherate in the presence of pentaerythritol.

Q. The first paragraph under section c says that since you got—I am paraphrasing—since you got the hard, brittle polymers using a large excess of propylene oxide compared to the amount of pentaerythritol, that the reactions between propylene oxide polymer and diisocyanate was studied.

It doesn't say anything about any pentaerythritol there. The next paragraph, you go on and you say five-tenths grams of sodium was added to 40 cc of propylene oxide and the mixture warmed gently; the reaction mixture after 20 minutes of reaction and the brown solid polymer which formed gave hard, brittle polymers when treated with diisocyanate.

Where is the pentaerythritol in that reaction? A. I don't recall running any reactions without the pentaerythritol present.

Q. Doctor, I am just reading your own report and that's what it says. A. Because what I was doing here, I was—this whole series of experiments was designed to study the molecular ratio of pentaerythritol to propylene oxide because what I was (402) trying to find out was how much cross linking here and network pattern did I need to achieve a rubbery product and the only way I know how to do that, following Dr. Price's directions, was to add various molar ratios of propylene oxide and pentaerythritol in the presence of a catalyst.

Q. Will propylene oxide polymerize with itself in the presence of sodium? A. When you add the sodium to the propylene oxide—let me say this: I don't recall adding sodium as the only catalyst or reactant with propylene oxide.

Q. That really wasn't what I asked you.

I asked you if propylene oxide would polymerize with itself in the presence of sodium. A. I didn't carry out any experiments to prove or disprove that point.

Q. Which means you don't know? A. My experiments—the purpose of my experiments was to put the pentaery-thritol into the reaction mixture and condense it with the—

The Court: Dr. Herbst, the question, I think, that Mr. Sweeney is entitled to answer to, as I understand it, was not involved in what your experiments were but he is asking a question as to whether, based upon your knowledge, I suppose, as to whether the propylene oxide will (403) polymerize with itself in the presence of sodium. That is his question.

The Witness: Would you restate that, please? What are the conditions? Specify the conditions of the experiment; under what conditions.

Q. Let's have it in atmospheric conditions. A flask, just the way you made your condensates of pentaery-thritol with propylene oxide. A. I don't know if that would happen under these specific conditions. I never tried it in a laboratory.

All my experiments were done in the presence of pentaerythritol.

Q. You said that. I appreciate that.

Do you know any way that you can polymerize propylene oxide with itself using sodium? A. Not using sodium with other catalyst systems which were, of course, developed a lot later than this one.

Q. Now, under Section c of your report, Plaintiff's Exhibit 20, you do not mention making a propylene oxide pentaerythritol polymer, do you? A. Where is this?

Q. Look under Section c on page 2 of Exhibit 20 and if you would, you tell me where it says that you used pentaerythritol in making the polymer that you reacted with the isocyanate. (404) A. I see no mention here in paragraph c of the pentaerythritol but let me answer your question this way: In the—

Q. I think you have answered my question. A. The-Mr. Sweeney: Your Honor, I believe the witness answered my question. I don't think I need a

speech.

The Court: I think that you have answered the question, doctor, and I think that I understand the point. I think you already indicated that you thought it wasn't mentioned, that you always experimented with pentaerythritol. You always used it. That's what you testified before.

The Witness: Yes, your Honor, we knew that by reacting sodium with the propylene oxide we were going to get hydroxyl groups present. We weren't sure that we were going to get a large or a high enough concentration of hydroxyl groups to build up this network.

The Court: All right.

Q. In any event, whatever you did in Section c on page 2 of Exhibit P. 20, you got about the same results that you got when you ran the reaction under paragraph h of that same page, correct? A. Again, I got similar results in that—in that with the diethylphthalate, I produced a rubbery material.

Q. And without it, you got- (405) A. Without it, I got--

Q. A hard, brittle polymer? A. A hard, brittle polymer; a brown solid polymer.

Q. All right. If you would, then, look at Exhibit P. 22. I just want to confirm with you that this report deals with the polyester work, has nothing to do with polyurethane work? A. This deals with the purification that I referred to earlier for the molecular weight determination. And also, under paragraph 3-the polyester work is described in paragraph 3 on page 2 of Exhibit 22.

Q. You say here in paragraph 3 of page 2 that—you list the compounds which gave the best rubbery characteristics. Those are all polyester compounds, aren't they! A. On page 2, paragraph 3?

Q. Yes. A. These are all polyester.

Q. And you considered them rubbery. Can you tell me how rubbery? A. They were pliable in the hand, flexible material, pliable, but also soft to the touch.

Q. Is this much the same as the other material you described, that you couldn't hall it up? A. Yes. These polyester products I could not ball up (406) and they would not bounce.

Q. Did you consider the polyurethane materials that you had made to be better rubbery materials than the polyester materials? A. Depending upon your—the intended use. I regarded the polyurethane materials better from the standpoint they were, they had this bounce, they were bounceable.

However, at the time T also felt that the rubbery polyester products would also have utility, too, as rubbery coatings and impregnates, not necessarily as a rubber tire or a rubber ball.

- (407) Q. Let's look now at Plaintiff's Exhibit 24 which is entitled "Summary Report." First of all, there has been testimony by Dr. Price and I think by you now that the reports that we have before us, Plaintiff's Exhibits 18, 20, 22 and 24, are the only reports that you wrote! A. Yes, sir. That's correct.
- Q. Then would you please tell me— A. My best recollection.
- Q. Please tell me what the report is that is referred to at the bottom of page 2 of Plaintiff's Exhibit 24 where it says "see research report, August 29, 1949." A. Where is this?

Q. Look at Plaintiff's Exhibit 24, the summary report, look at page 2, the very bottom of the page that says "see research report, August 29, 1949." A. I don't recall that report.

Q. You don't know what it was? A. I have no recol-

lection.

Q. You typed this document yourself, did you not? Exhibit 24. A. Exhibit 24, yes. Yes, I did.

Q. Do you have any idea where that report might

be? A. No, I do not, sir. I don't know.

- Q. All right, I want you to look now at page 3 of the (408) summary report, Plaintiff's Exhibit 24. I don't wish to be repetitive because Dr. Price has already testified about this, but the middle paragraph where it says "reactions between PO PET polymers and diisocyanate" is simply a report of what you had done before, am I correct? A. Yes, sir. This was not—my best recollection is this is not any new information. It was an attempt to describe a typical reaction here to make the polyurethanes. This describes the small scale experiments. It does not describe the scale up experiment where I made the larger balls.
- Q. As a matter of fact, those are not described anywhere, are they? A. Not in this documentation but they certainly were described in my notebook.

Q. The missing notebook? A. Yes, sir.

Q. In the last phrase of the paragraph that I referred you to on page 3, you say "only slight improvement resulted in the 'rubbery' properties of the polymers." You are referring there to the reaction in a solvent.

Why is it if you made such beautiful rubber balls you put in your summary report that only a slight improvement resulted in these rubbery properties? A. First, let me correct one thing. I did not call them (409) beautiful rubber balls, sir. I called them rubbery balls. They were an improvement in the sense that they were bounceable when carried out in the presence of the solvent.

- Q. In other words, what you mean here is that only a slight improvement over the hard brittle polymers was obtained when you carried the reaction out in a solvent? A. Yes. In terms that the materials made in the solvent were bounceable, whereas the polyurethanes made in the absence of solvent were not bounceable, they were hard materials.
- Q. These are the ones that you made in the diethylphthalate and the alphachloronaphthalene? A. That's correct, sir.
- (410) Q. If you would, look at page 4 of that same report, Doctor, Plaintiff's Exhibit 24. I want you to look at the conclusion of the section down at the bottom, and in the second paragraph of the conclusion section you say, "The best rubbery polymers are formed by the reaction of the POPET polymers with maleic anhydride," etc. Those are all polyesters, aren't they, Dr. Herbst! A. Yes, and they are all carried out in the absence of a solvent.
- Q. And the reaction—then you say that the reactions with the diisocyanate produced hard brittle polymers. A. In the absence of solvent.
- Q. I don't see the words "in the absence of solvent." (411) A. But this whole series of experiments was done in the absence of solvents, as I indicate.
- Q. What whole series? A. The whole series on polyesters. I was simply comparing the various polyesters at the top of page 4, in that table there.
- Q. I want you to point out to me how I can be sure that's what you are doing. A. Let me explain in this way. Dr. Price asked me to write up in here additional experiments that had not been written up previously, and this is what I included in this particular wrapup or final report, and its main emphasis is on the polyester work.

Q. As a matter of fact, the polyurethane work stopped some time in the middle of the summer, didn't it? A. No. The Polyurethane work continued, but along the emphasis of making the larger ball for Professor Price.

Q. Can you tell me why in the conclusion paragraph you don't even mention the rubbery properties of the urethane materials that you made? A. There was no need to. He asked me not to repeat what I had told him earlier and he had been critical in the past of my wordy and lengthy reports, and this was an attempt to cut down on the wordage.

(412) You will note the other conclusion at the last sentence, though, I did indicate that in the future one should strive to obtain the higher molecular weight polymers, the high molecular weight propylene oxide pentaery-thritol polymers, because I felt at the time that this would be the direction to take to achieve the better physical and mechanical properties.

Q. You did not do it, though, take this direction? A. At this point I left Notre Dame. However, I did feel I had in a practical way taken Professor Price's ideas, his concept of taking a volatile liquid containing oxygen and converting that by reacting it with sodium pentaerythritol and then reacting that product with a diisocyanate and also with dibasic acids anhydrides to make rubbery materials. This was accomplished.

We did not accomplish, certainly, an ultimate objective of making a rubber tire. No, we did not do that, but we did make giant sized molecules quite insoluble and which were rubbery.

Q. Look at the top of page 4 once more. You say there "maleic and succinic anhydrides gave the best rubbery material. You repeat that practically the same way in your conclusion, don't you! A. This refers to the polyester work.

Dr. Ralph L. Herbst, for Plaintiff, Cross

Q. Right. But you don't repeat any of your conclusions (413) about the polyurethane work being rubbery. A. I had already reported that in my very first report and had made the rubber balls for Professor Price, had made some larger tennis balls and the—this effort in the latter part of the project was directed towards expanding the scope of this chemistry and there was more emphasis put on the polyester part of the work in the last several months of the project.

Q. How large a quantity of material did you use in the pressure bomb that you have talked about? A. This, I can only describe the dimensions of the bomb, it was a stainless steel cylinder about that round and about

that high.

Q. It's awfully hard for the court reporter to pick that up. I wonder if we couldn't agree that that's about the size of a thermos bottle or something of that nature? A. Yes. It was about the size of a pretty, you know, large sized thermos bottle, a larger sized thermos bottle.

Q. Would you make a guess as to the contents—the size of the—what the contents' size might be? My question

is a little gummed up. I will try it over.

How much liquid would the bomb hold? Let's put it that way. A. I believe it would hold about a quart if it were (414) filled to the top. As I recall, though, I was very careful not to fill it to the top because I was getting pressure. I did not want to—I wanted to leave some space in there to handle the pressure.

So I do recall that I did not fill this all the way up to the top. I left some space between the top of the

liquid and the top of the bottom.

Q. Earlier I think you testified that you thought the example 3 came from the—of the Price patent—came from the scale up work that you said you had done. A. Yes.

Dr. Ralph L. Herbst, for Plaintiff, Cross

· Q. And that the amount of 15 grams of TDI came from that work. A. Yes, sir. That was done not, though, from the—that was not done from the work carried out in the pressure bomb.

Q. I wasn't trying to allude to that. I changed subjects. I am just talking about example 3 of the Price patent that you said that came from your scale up work.

(415) By Mr. Sweeney (con't)

Q. Talking about example 3, we are back to that, Dr. Herbst— A. All right.

Q. The 15 gram amount of TDI that's given there, where did that come from? (416) A. This came from the scale up experiments I did using the procedure I described in my first report, Exhibit 18. The one here, paragraph 2, a reaction at room temperature for three days followed by heating at 110 to 115 degrees. My initial experiments were carried out in the test tube scale, as I have testified earlier.

When Professor Price requested that I make a larger tennis ball, then I repeated this work in a larger scale. Both in making the propylene oxide pentaerythritol condensate, but in a larger scale reaction, in a larger flask, in a range of 100 to 125 milliliters, where I was reacting the diisocyanate with the propylene oxide pentaerythritol condensate.

- Q. We have it straight now that you have testified that that's where the 15 gram amount of the TDI came from? A. It was in those scale up experiments which I just described, yes, sir.
- Q. When I took your deposition, on page 65 I asked you, referring to this same area:
- "Q. And in that same sentence it mentions 15 grams of TDI. That is not in the report either, is it? A. That's not in this report, no.

Dr. Ralph L. Herbst, for Plaintiff, Cross

"Q. Do you know who did that work, in other words, the mixing of the 100 of the polymer with the 15 grams of TDI? A. No, I do not."

(417) A. I don't know who wrote up this example in the patent, sir.

Q. That isn't what the question was in this deposition, Dr. Herbst. The question was who did the work. You said then you did not know. You say now you did. A. Yes, I did do it and there was no one else, I'm the fellow who did the work in the scale up, but I did not write up this example in the patent application. The example 3.

(452) Colloquy

Mr. Blecker: May I have Plaintiff's Exhibit 41? Your Honor, I would like to point out that Mr. Ries was a very major part of the negotiations between Jefferson and Mobay; in these negotiations we say this was all part of the picture of the misuse.

The Court: I understand that.

Mr. Blecker: So, he is a fact witness as well. However, we are trying to cooperate in this respect. I would like to point to Plaintiff's Exhibit 41, which is a memo from Mr. Ries in which he refers to two Heiss patents. If Mr. Sweeney is willing to stipulate that the patent in the second paragraph referred to as the first Heiss patent is the Heiss patent in issue in this case, perhaps we could avoid the problem of examining Mr. Ries at this time.

That second paragraph, the portion I am referring to is in the middle of the paragraph. It says: "As to the first Heiss patent, I indicated that there is a doubt as to its validity and to the pro-

priety of approaching Jefferson rather (453) than the foam manufacturers."

Mr. Sweeney: May I have just a minute?

The Court: All right.

(Pause.)

Mr. Sweeney: I gather you want me to agree that the patent that is referred to in the second paragraph is the Heiss patent in suit?

The Court: That is correct.

Mr. Blecker: Yes.

Mr. Sweeney: Yes; that is correct.

Deposition of JAMES HENRY SAUNDERS (455)taken on January 19, 1972 at the Monsanto Company, 800 North Lindberg, St. Louis County, Mis-

(Pages 3, line 7 through page 6, line 2.)

"Mr. Kosinski: Now, Dr. Saunders, would you please state your full name?

"A. James Henry Saunders.

"Q. What is your present home address? (456) A. 4151 Piedmont Road, Pensacola, Florida.

"Q. What company are you presently employed by?

A. Monsanto Textiles Company.

"Q. In what capacity? A. Director of Nylon and Polyester Research.

"Q. How long have you been employed in that posi-

tion? A. Since December of '69.

"Q. Were you employed by Monsanto prior to December of '69? A. Yes. I was manager of Nylon Research from about March 1, 1968 to about December of '69, the same location.

- "Q. What was your employment and position prior to March 1 of 1968? A. I was with Mobay Chemical Company in Pittsburgh, Pennsylvania. I was Director of Research for them.
- "Q. What was the period of your employment with Mobay? A. I joined Mobay approximately when it was formed, about July, 1954, and continued with them until 1968.
- "Q. Now, would you briefly indicate the various positions which you held at Mobay during the period July, '54 through March 1 of 1968? A. I was Assistant Director until 1959 and then was Director of Research after 1959.
- "Q. Were you Assistant Director from January, '54 (457) until what date again? I mean July '54 to what date? A. Approximately that time until approximately May, 1959.
- "Q. And then you were Director of Research from that date until you terminated your employment with Mobay, which was approximately March 1 of 1968? A. Yes.
- "Q. Now, what was the nature of your employment prior to July of 1954? A. I was employed by Monsanto at Anniston, Alabama as a chemist and then as a group leader in research.
- "Q. When did you commence your employment with the Monsanto Company? A. July, 1947.
- "Q. Prior to developing further your various duties and capacities with Monsanto, commencing in July of 1954, would you briefly describe the educational background after high school? A. I received a Bachelor's degree in Chemistry from the University of Kentucky in 1944; Ph. D., Organic Chemistry, University of Illinois, 1946; one year of post doctorate research at Illinois, '46 to '47.

"Q. Now, did your postgraduate research work include any extensive work in the field of polyurethane chemistry? (458) A. No.

"Q. What was the nature of your doctoral thesis, then, at the University of Illinois which was submitted in partial fulfillment of your Ph. D. degree? A. Synthesis of Unsaturated Monomers and Polymerization to SBR Type Polymers.

"Q. What is an SBR type polymer? A. Styrene-

butadiene.

"Q. When you commenced your employment at Monsanto in July of 1954, do you recall the nature of your first assignment, your first employment assignment? A. I started in '47, is that the date you're referring to?

"Q. Yes, '47. That's right. A. The initial assignments

were in the area of preparation of isocyanates.

"Q. Now, how long a period of time were you engaged in the work on the preparation of isocyanates? A. Off and on for the period 1947 to 1954, with Monsanto."

(Page 6, lines 19 through 23.)

"Q. All right. Did there come a time when you conducted research work in the field of polyurethane products, which are reaction products of polyisocyanates with compounds (459) containing active hydrogen groups? A. Yes."

(Page 12, lines 17 through 24.)

- "Q. I see. Now, do you recall independently the approximate time that Mr. Heiss commenced his employment at Monsanto? A. About 1951.
- "Q. About 1951. To what group was Mr. Heiss assigned when he commenced employment at Monsanto? A. Mine.
 - "Q. Did he report directly to you? A. Yes."

(Page 30, lines 18 through Page 31, line 15.)

- "Q. Do you recall what was the position of Mr. Rucker at Monsanto during the period of 1951 to 1953? A. He was the patent attorney.
- "Q. Was he the only patent attorney at Monsanto during that period? I'm speaking of the Anniston research facility. A. As far as I remember, he was the only one at Anniston.
- "Q. What was the practice which prevailed at the Anniston research facility at Monsanto in 1951 to 1953 in connection with the submission of invention disclosures by you or any other personnel who worked in your group? (460) A. We submitted information to the—to Mr. Rucker, the patent attorney, along with any pertinent information we knew of concerning the literature. He decided what the invention was, if any, and from that established who the inventors were.
- "Q. In what form was this application submitted? In other words, was it orally or was it in the form of a letter to Mr. Rucker, or— A. As I recall, it was a combination of written information followed by oral discussions."

(Page 59, Line 24 to page 61, line 3.)

- "Mr. Kosinski: Dr. Saunders, I hand you a document marked Monsanto Deposition Exhibit 3 (P-28) which bears the date of June 21, 1954, and ask you if you received this letter from Mr. Rucker on or about the date indicated, namely, June 1, 1954? A. I don't recall specifically seeing it. I have no reason to think that I did not.
- "Q. Does that document refresh your recollection as to whether you received a copy of the office action which appears on page 27 of Heiss Exhibit 41 (P-2) on or about June 21, 1954? A. I don't recall receiving it specifically.

"Q. Do you have any reason to believe that you did (461) not receive it as indicated in the letter marked Monsanto Exhibit 3 (P-28)? A. No.

"Q. Do you recall considering any of the references which are cited in the office action which appears on page 27 of Exhibit 41 (P-2) on or about the date indicated in Monsanto Exhibit 3, namely, on or about June 21, 1954? A. I don't recall.

"Q. Now, I again hand you Monsanto Deposition Exhibit 3 and ask you if that is your handwriting which appears on the lower portion of this Exhibit? A. Yes.

"Q. It starts off with the name "Elmer" and it's signed "Jim." Was this a note which you wrote to Elmer P. Rucker in response to his letter to you set forth in the top portion of Monsanto Exhibit 3 (P-28)? A. Yes.

(Page 62, lines 7 through 18.)

"Q. Dr. Saunders, I hand you a document which has been marked Heiss Deposition No. 28 (P-29), and ask you if that is your signature which appears on the bottom of the first page of that document? A. Yes.

"Q. Did you prepare— (462) A. Excuse me. I'm not

sure I heard all your question.

"Q. Did you prepare this entire document, namely, the subject matter which appears on pages 1 through 3 thereof? A. I wrote it. I doubtless got the list of notebook pages from someone else as a tabulated list. But other than that, yes."

(Page 63, lines 2 through 8.)

"Mr. Kosinski: For what purpose was this document prepared?

"A. To report the work which had been done.

"Q. As far as you were aware, this contained an accurate summary of the work which had been done on this particular project? A. Yes."

(Page 104, lines 11 through 24.)

By Mr. Sweeney:

"Q. There has been a lot of talk about Aroclor during your testimony here today. I think it would be wise if we clarified just what Aroclors are. Can you tell us that? A. Aroclor is a Monsanto tradename describing any one of a group of chlorinated biphenyl compounds.

"Q. Can you give us a little more information on them, without divulging any proprietary information, of course? (463) A. These materials have been used commercially with excellent results as liquid dielectrics and head transfer media.

"Q. Are they also solvents? A. They are solvents for certain products. As far as I know, that is not a significant commercial use."

(Page 114, line 22 through page 115, line 20.)

By Mr. Sweeney:

"Q. Dr. Saunders, I'll hand you back this Monsanto Exhibit No. 3 (P-28) and ask you to look at your handwritten note on the bottom, particularly the last sentence or so. A. All right.

"Q. Now, may I just look at it for a second so I can ask you a question? You're telling Mr. Rucker to delete your name, I gather, from the jelling of the compatible organic liquid development. What did you mean by that? A. I said I thought we had two novel points in the patent application. One was making foams from urethanes and isocyanates with a strong catalyst and the other was jelling the compatible organic liquids, for example, Aroclor. I asked if it would be appropriate to refile covering those separately and specifically, and then I said, 'If so, delete my name on the application on jelling the liquid.' I felt that I was not a co-inventor on that (464) specific part of the invention if there were an invention.

"Q. You mean a co-inventor with Mr. Heiss? A. Yes. "Q. But that you felt you were an inventor in connec-

tion with the other development? A. Yes."

(479) Interrogatory 24

State whether defendant makes, uses or sells any commercial product or uses any commercial process which is covered by any claim of U. S. Patent No. 3,102,875. If the answer is in the affirmative, identify each such product or process, designate the claim or claims of said patent alleged to be covered by such product or process and state the earliest date when such process was used or such products were made, sold or offered for sale.

Answer-filed 7/22/71

Jefferson is not in the business of producing polyether urethane products and assuming that "covered by" means in direct infringement of the claims of the patent, the answer is none.

(497) PHILTP MONAGHAN, called as a witness by the defendant, being first duly sworn, testified as follows:

Direct Examination by Mr. Sweeney:

Q. Where do you reside? A. Houston, Texas.

Q. By whom are you employed? A. Jefferson Chemical Company, Incorporated.

Q. How long have you been with them? A. Since 1950.

Q. What positions have you held over that period of time? (498) A. Initially as a technical service man; then manager of technical service; then Eastern Regional Sales

Manager; and, then, General Sales Manager; and, then, General Manager of Marketing and, now, Vice-President of Marketing.

Q. Based on your experience, could you please describe to the Court the American urethane industry as it is known today? A. It's an industry that is approaching a half a billion dollars of sales and in pounds, it's something in the order of magnitude of a billion pounds a year and it is, as you described earlier, Mr. Sweeney, used to produce products for cushioning, carpet underlay—that's the flexible material; it's used in automotive construction for crash pads, arm rests, seating, the visors.

It is used on some humper material in the elastomer form. The material is used to make insulation. It is also used as a wood substitute, as illustrated by that one piece.

And there are some coatings and caulks and sealants and so on. By and large the largest part of the billion pounds is in the form of flexible and rigid, with flexible being larger than rigid.

It is also a very fast growing business. The growth rate on the flexible urethane foam is probably in (499) the order of 15 percent per year and growth rate on the rigid urethane foam is in the order of 20 or 25 percent per year.

- Q. You said that there was more flexible foam sold than rigid. Do you know the proportions? Just round numbers. A. Oh, I would say about two to one.
- Q. What business is Jefferson Chemical Company in? A. In the chemical business.
- Q. Can you describe it generally? A. We make derivatives of ethylene and propylene. I should really say ethylene oxide and propylene oxide, including the polyols that go into the manufacture of urethane foam.
- Q. Historically, when were the first urethane foams used in the United States? A. The technology was, in the

commercial part of it, was pretty much imported from Germany, to some extent from England, as World War II technology.

Q. What kind of technology was that? A. Well, it was the polyester type of urethane, initially. That is to say, glycols reacted with adipic acid to make polymers which in turn were reacted with diisocyanates to get a urethane.

(500) Q. Whereas the polyether foam— A. The polyether foam didn't come along until somewhat later and it was significantly more successful than the polyesters for a number of reasons:

First of all, it was economically more attractive for at least two reasons: One, the polyester polyol was more costly to produce and, secondly, the ratio of isocyanate to polyester—I'll rephrase that—more of the isocyanate was required to make a urethane foam when you used a polyester polyol and the isocyanate is a much more expensive part of the molecule than the polyol is.

So, commercially, there was a driving force to go toward the foam that used less isocyanate and the polyether polyol did that because you were able to make bigger

molecules, higher molecular weight molecules.

In addition, as I think has been mentioned in the court, the performance of the polyether polyols was superior in several respects, particularly with respect to being subject to hydrolysis; that is the foam would break down, the polyester foam would tend to break down by a hydrolysis reaction, just from atmospheric moisture, but the polyether urethanes were, are quite impervious to hydrolysis.

The Court: So that I am clear about this, would you give me the properties of the polyester polyols and (501) polyether. I think I know what they are.

The Witness: In terms of the foam, your Honor, you really couldn't tell much difference. We are able to make

foam of lower density with polyether polyols than with polyester polyols but for the same density, you wouldn't really be able to tell the difference.

The Court: There are different products? That is what

I wanted to know.

The Witness: The differences lie in the stability of the polyether urethane foams being greater than the stability of the polyester urethane foams.

The Court: Are they the same properties, both made

with the propylene and the-

The Witness: No, sir. The polyester is made with a dibasic acid, like adipic acid and a glycol like diethylene glycol.

To my knowledge, we have not made the polyesters using propylene glycol and I won't try to comment other than that.

Q. I think perhaps what the Court was asking was, the polyester foam is made differently than the polyether foam. A. That's correct, and it's a more expensive process to get the foam using the polyester approach.

Q. And the components in the polyether foam are, at (502) least the ones we have talked about, the propylene

oxide based triols? A. Correct.

Q. And diisocyanate? A. Correct.

Q. Whereas in polyester, it's a polyol and acid and an isocyanate.

The Court: All right.

Q. Are you aware that a patent was issued in 1958 to Dr. Price? A. Yes, I am.

Mr. Sweeney: That is marked as Defendant's Exhibit A.

I'll ask the clerk to mark this document as Defendant's Exhibit S for identification.

Mr. Kelton: No objection.

(Defendant's Exhibit S received in evidence.)

Q. Mr. Monaghan, can you identify Defendant's Exhibit S? A. Yes.

Q. Would you tell the Court what it is, please? A. It's the license agreement between Jefferson Chemical Company and General Tire to have the right to practice under the Price patent.

Q. Do you know how much Jefferson paid for that li-

cense? (503) A. Yes. \$200,000.

Q. Was Jefferson the only licensee? A. To my knowledge, no.

Mr. Sweeney: Judge Brieant's order which I had marked in lists the number of licenses that were granted and I don't choose to go into it with this witness, since it's been agreed that they were substantially the same as the Jefferson license; at least that's what Judge Brieant's order says.

The Court: You mean that the licenses by General Tire were granted under the Price patent to

several other companies.

Mr. Sweeney: That's right.

The Court: Other than Jefferson?

Mr. Sweeney: That's right, and they were all substantially the same.

Q. Mr. Monaghan, I am sure you know this case involves the Heiss patent.

I would like to ask you, does Jefferson own that patent? A. Yes, we do.

Q. How did you come to buy it? A. We purchased it from Mobay.

Q. Do you recall the circumstances of how you came to (504) buy it? A. We were approached by Mobay. They had several patents they wished to sell. We considered these matters and felt that we had an obligation or a liability under the Heiss patent and we also felt that the Heiss patent could be a potential source of revenue to us.

- Q. What did you do with the patent after you purchased it? A. We attempted to license it.
- Q. Can you tell the Court how you went about attempting to license it? A. Well, we made it known to the public and to the industry that we had acquired the patent and we had, I guess you would say, offers extended to the other polyol manufacturers which was a practice that had been followed by General Tire in their price licensing program.
- Q. Was a licensing form ever prepared by Jefferson under the Heiss patent, a form of license? A. I believe there is in—the record shows that, does it not?

(505) By Mr. Sweeney (cont'd)

- Q. Mr. Monaghan, can you identify Defendant's Exhibit T for identification? A. Yes, I can.
- Q. Would you tell the court what it is, please. A. It's a draft of a proposed agreement to the parties, wherein Jefferson would license them to practice the Heiss (506) invention.
- Q. Do you know whether that agreement was ever delivered to any potential licensee? A. I personally delivered one to WitCo.
- (508) Q. Now, Mr. Monaghan, did your sales people play any role in the attempts to license? A. Yes.
- Q. Can you tell the Court what they did? A. Well, they were informed of our having acquired ownership of the patents and then a memorandum was issued to them relative to our contacting polyol producers to offer licenses and they were informed on these matters.
- (510) Q. Did Jefferson issue a press release relating to its acquisition of the Heiss patent? A. Yes.

Philip Monaghan, for Defendant, Cross

Mr. Sweeney: I ask that this matter be marked as Defendant's Exhibit Y.

(Defendant's Exhibit Y marked for identification.)

- Q. Mr. Monaghan, I hand you a copy of Defendant's Exhibit Y and ask you if you can identify it as the press release that was issued by Jefferson. A. Yes, I can identify it as being the press release.
- (511) Q. What was the purpose of the press release, Mr. Monaghan? A. Well, it was an inexpensive way to communicate to the public that we had made this acquisition.

Q. Did the information in the press release appear

(512) in any of the trade publications? A. Yes.

Q. What was the response to your attempt to license the Heiss Patent? A. Totally unsuccessful. By and large, people felt that they had already paid General Tire to practice the same technology and they didn't think it was right to have to pay a second time, so to speak.

Cross Examination by Mr. Blecker:

(519) Q. I call your attention to paragraph 4.2 of Plaintiff's Exhibit 30, which appears at page 5 of the agreement and I refer you to a term that is mentioned in the agreement, "polyoxy-alkylene polyether polyols".

It refers to a manufacturer of polyoxy-alkylene poly-

ether polyols.

Could you describe what that means? A. What what means?

Q. What the term, "a manufacturer of polyoxy-alkylene polyether polyols." means. A. The term manufacturer is obvious. Polyoxy-alkylene polyether polyols are the polyols that we have been talking about here today and yesterday.

Philip Monaghan, for Defendant, Cross

They are reaction products of propylene oxide and a polyol—glycerine, pentaerythritol.

(521) Q. Mr. Monaghan, is it or was it your testimony that the polyoxy alkylene polyether polyol manufacturer is one that reacts propylene oxide with an alcohol? A. Yes, that's correct, an alcohol or a glycol or a triol.

Q. That reaction would be, then, a reaction of an alky-

lene oxide and triol or a diol? A. Correct.

(524) By Mr. Blecker (cont'd)

- Q. Mr. Monaghan, did you grant any licenses at all under the Heiss patent? When I say you, I mean Jefferson. (525) A. No.
- Q. Did you grant any licenses to your customers? A. No.
- Q. Did you make any efforts to license foam manufacturers as distinguished from polyol producers? A. Yes.

Q. Who? A. General Tire.

(527) By Mr. Blecker (cont'd)

Q. If you will turn to the second page of the exhibit, it mentions as one of the purposes of purchasing the patent that it was Jefferson wanted to protect its present heavy commitments in polyol and catalyst production.

What did you mean by that? A. The Heiss patent, of course, involves the use of polyols and we wanted to free ourselves of any encumbrances under the Heiss patent

by owning it,

The catalyst production I believe relates to some of the other patents. That is to say, some of the other (528) patents acquired by Mobay had to do with a certain amine catalyst which Jefferson manufactured and sold.

Q. So the reference to the catalyst was the reference to the other patents that were involved in this acquisition; is that correct? A. Yes; patents and application. You remember there is one application.

Q. But was your reference to the heavy commitment in polvol production a reference to the Heiss patent?

Was that why you purchased the Heiss patent, to protect the polyol production? A. Well, first of all, we don't say a heavy commitment in polyol production. We say a heavy commitment in polyol.

I'm sorry; we do say production.

Our intent there is that we have a commitment that is broader than just steel and concrete.

We have a knowledge, a body of knowledge that was very, very costly to develop; the commitment is a very large commitment totally. That's what we were referring to.

(532) HERBERT L. HEISS, being first duly sworn, was examined and testified as follows:

Direct Examination by Mr. Sweeney:

Q. Where do you live? A. New Martinsville, West Virginia.

Q. Would you give the Court a brief summary of your educational background, please? A. I received a Bachelor of Arts Degree from Lawrence College, now called Lawrence University, in Appleton, Wisconsin in 1943.

Then, after I got out of the Service, out of the Navy, I took a refresher course at the University of Illinois for about two semesters and that is the extent of my formal education.

Q. Since leaving school, what has your professional occupation been? A. A research chemist.

Q. By whom are you and have you been employed? (533) A. B. F. Goodrich Company, S. C. Johnson & Son, Monsanto Chemical Company.

Q. By whom are you now employed? A. Mobay.

Q. What have your duties been at Monsanto and Mobay, Mr. Heiss? A. I started out as a research chemist and then I went to a research specialist and at present I am the senior group leader.

Q. Any particular field of research? A. It has all been

with urethane chemistry.

- Q. Why did you leave Monsanto and go to Mobay? A. When Mobay was formed, the people who had been associated with isocyanate work in Monsanto were automatically transferred to Mobay.
- Q. Maybe you had better explain what Mobay was formed from? A. Mobay was a joint venture by Monsanto in this country and Farbenfabriken Bayer in Germany, who had the main technology in the isocyanate urethane technology at that time.
- Q. When did you first start to work for Monsanto? A. 1951, about March.
 - Q. Pardon? (534) A. About March.
- Q. Where were you geographically located? A. Anniston, Alabama.
- Q. What project were you assigned when you went to work for Monsanto? A. I was the first man they hired to work on the application of isocyanates at that location.
- Q. Why was Monsanto interested in finding applications for isocyanates? A. They were making two isocyanates at that time and were interested in expanding the field of technology.
- Q. What did you do in looking for new uses? A. Well, about that time some of their PB reports and BIOS reports were coming over from Europe, from Germany, and we knew that the urethanes were used over there in foams, in elastomers, in coatings and in adhesives and we then

tried to see if we could help that industry get established over here.

Q. This was the polyester urethane? A. Yes, the Bayer products, the German products, are all based on polyesters.

Mr. Sweeney: Your Honor, you are going to notice with Mr. Heiss, as I think you might have with Dr. Price, chemists tend to talk in the editorial "we" and I have (535) asked Mr. Heiss not to say "we" when he means I, but he slips quite frequently.

The Witness: Yes, I slip.

- Q. What materials did you try, Mr. Heiss? A. For what?
- Q. When you started your application work with the isocyanates? A. The first assignment we had was to work on isocyanates splitters and I tried a series of alcohols. The purpose of this was to make products where the isocyanate group was completely reacted away, but then on further heating it would regenerate isocyanate.

That wasn't worked on too long until some of our sales people decided there might be more future in working on adhesives, so at that point we started or I started getting involved in the polymers.

Q. What did you do in the beginning? A. Well, again since we knew of the German experience, where the polyurethanes were based on polyesters, it didn't seem very profitable to repeat their work, so we were trying to generate new information so I considered what other types of materials we could react isocyanates with to form polymers and I was familiar with the polyethers, polyols—well, polyethers, really, available at that time and so we ordered (536) some of them in and started working with them.

- Q. Did you do any work with diols? A. Yes, they were all diols at that time.
- Q. Did you react these materials with isocyanates? A. Yes.
- Q. What did you get? A. We got a variety of materials depending upon which diol you used. You could get anything from a hard glassy material to a soft rubhery material.
- Q. Did you evaluate these products at all at the time? A. Yes.
- Q. What was your evaluation? A. Well, we looked at them in several different ways, since we weren't aiming at any specific one type of product, we were just trying to see how many—how great a variety of products could be made from polyisocyanates.

The Court: Would you inform me—I can ask you directly—during this period I am not clear of the time.

Mr. Sweeney: All right.

The Court: When you were making these experiments. It would be helpful for me to know the years you were doing this.

The Witness: All right. I am talking still about 1951.

(537) The Court: All right.

Mr. Sweeney: Your Honor, I might say that I plan to put the notebooks in and the notebooks are all dates, the pages, and I am just doing a general summary, if you will, at the moment.

The Court: Fine.

- Q. Did you do any work with triols at this time? A. Initially, no. There were no triols commercially available at that time that I was aware of. A high molecular weight triols.
- Q. Did you perform any experiments using glycerine at this time? A. Yes.

Q. What was the result of that? A. If we used glycerine with an isocyanate alone, it was a very hard glassy brittle material, which was of not much value, but we could mix glycerine with some of these high molecular weight linear materials and get a variety of more useful products.

Q. Did you evaluate those products? A. Yes, again in all the different categories as foams and adhesives.

Q. What was the next step that you took in this research program? (538) A. Well, I could see there was some advantage to having branching in the materials when we added a low molecular weight glycerine, but I wondered if we couldn't possibly get a better polymer if we built the branch points into the high molecular weight material.

It is kind of hard to explain, but if you use glycerine, you have a branch of material which is very compact, it sort of looks like the letter Y and when these react together you get very tight crosslinks, as they call them, which make the material sort of rigid.

What I wanted was a material which was branched, but each leg of the Y was much longer before it.

To get a more rubbery material and that is what I wanted to try out.

Q. What did you do in attempting to try this out? A. I could not find any commercially available or even what they call pilot plant materials available in the industry so we had a group working at Anniston Detergents that were equipped to make ethylene oxide condensates, so I asked them if they could make for me a reaction product of some triol, like glycerine or TMP—

Q. You had better define what TMP is. A. Trimethylolpropane, to react with that.

The TMP or glycerine with ethylene oxide—we can (539) abbreviate that to EO and PO—to make this high molecular branch material and I found that they had al-

ready done that. Flores had a sample he had made previously of glycerine and EO, so I tried that first.

Q. What was the result of that test? A. It looked interesting and we could see from a polymer standpoint we got a more interesting polymer than when we mixed

glycerine with polyethylene glycol.

Q. Did you evaluate the product that you got from that reaction? A. Yes, and we made some foams, I believe, and some adhesives, but the weakness there, as with all the ethylene oxide products, was that they were hygroscopic, which means they pick up moisture and become plasticized and softer and so from a practical standpoint, for example, if you had a foam on a dry day it would become firm and on a damp day it would become soft; so that wasn't too practical, so we wanted something that was less hygroscopic than that.

Q. What did you do to overcome this problem? A. Well, I wondered what would happen if we used PO instead of EO. I went back to the detergents group, Mr. Satkowski was the group leader and asked him if he could make for us some condensation products of glycerine with

varying amounts of PO.

- (540) Q. What happened as a result of this request? A. He promised to do this when it could be worked into their schedule, but he pointed out they were in a surfactant business and it might be a while before they could work that in for us.
- Q. Did you, in fact, get this material? A. Eventually, yes.
 - Q. Who made it? A. Hector Flores.
- Q. Did you react that material with isocyanates? A. Yes.
- Q. Describe that reaction, if you will. A. The reaction went very nicely and we got a product which had all the benefits of the glycerine EO, in the sense of being a nice polymer, but in addition it was superior in not being

moisture sensitive. It did not soften to such a great extent by moisture.

Q. Did you keep research records of the work that you did? A. Yes.

Q. Did you have any people working for you on this project? A. Yes.

Q. Did they keep research notebooks? (541) A. Some did and some didn't. The technicians generally merely were a pair of hands. They did work and it was written in my book.

Other people, such as Bill Davis, were trainees who were eventually supposed to become technical people, and these people would keep their own notebooks.

Q. But you supervised all of their work, didn't you!
A. Yes.

Q. Are the notebooks that you have been testifying about here? A. Yes.

Mr. Sweeney: Mr. Clerk, I have marked the copies along with them with the proper defendant's identification number.

The first is Defendant's Exhibit Z. The second is Defendant's Exhibit AA, etcetera.

(Defendant's Exhibits Z, AA, AB, AC, AD and AE marked for identification.)

Mr. Sweeney: I might say, your Honor, that though you have got a lot of paper and there are whole books here, we really don't need the whole book; there are pages that we need. I am in hopes that once we get through with this we can agree to use the pages instead of all this paper.

The Court: All right.

(542) Mr. Sweeney: In any event, the Clerk has now marked defendant's exhibits Z, AA, AB, AC, AD, AE.

Q. Mr. Heiss, I hand those exhibits to you and ask you to tell the Court what those books are. A. Book number Z is one of my notebooks.

AA is one of my notebooks.

AB is one of mine.

So is AC.

So is AD.

AE is the notebook of Bill Davis.

The Court: Mr. Davis was one of the people who worked under your supervision?

The Witness: Yes, sir.

Q. I would like you to look first at page 73-323 and, if you will, that is in defendant's exhibit Z and tell the Court what work you did as reported on that page? A. On that page is entitled ABL project.

Q. What is the date of that, Mr. Heiss? A. That is

September 4th, 1951,

ABL stands for alleghany Ballistics Laboratory. They came to us with an adhesion problem in conjunction with rocket propellants and we were trying to prepare for them a special adhesive.

On this page are some of the first attempts where (543) we reacted TDI with varying use low molecular

weight linear polymer glycols.

Q. What further happened with that reaction, Mr. Heiss? A. We had a little problem with compatibility on the polyethylene glycol series, although they did clear up after reaction, but the polypropylene glycol series were more compatible with the isocyanate and we were able to prepare a series of prepolymers. That is all that was done on this page.

Q. Can you describe the products that you made on September 4th, 1951; what did they look like? A. Well, it says on the following page, 24, that they were all either viscous liquids or glasses at room temperature ex-

cept E, which was an opaque white solid.

Q. What did you do with those products? A. What we wanted to do with them was to use them as adhesives. These were materials which were of moderate molecular weight and which were terminated with NCO groups and we wanted these NCO groups to begin reacting with atmospheric moisture so that our adhesive would polymerize or cure and change from a liquid into a tough elastomer, which would act as an adhesive.

Q. Now, if you will, look at page 73325, which is in (544) defendant's exhibit Z. A. Well, the other materials were linear. They had only two OH groups per molecule. On this one we tried to see what would happen with 3 mols of TDI with one mol of glycerine. We got a very exothermic reaction. It was sort of an incompatible mixture. It wasn't a very good reaction, really.

Q. What did the product you got look like? A. Well, it became more and more viscous, but it didn't remain clear like some of the others did and some of the products started precipitating out and it ended up as a cream colored opaque solid mass. It was a dispersion, really.

Q. Now, would you look at page 73334, which is in exhibit Z. A. This was dated September 26 to October 3rd. There are a series of entries on this page, 1951.

Here we tried a more complicated molecule and we tried to react TDI with a pentaerythritol. That was really bad. The pentaerythritol wasn't very soluble with TDI. Again we got a liquid viscous reaction product. This was on September 27, but apparently the reaction wasn't over, it continued to react.

I see entries here on the 28th where it says unreacted pentaerythritol is still present and on October 3rd (545) I see another entry where we apparently gave up on this candidate because we say, "Due to the apparent

unsuitability of TDI adducts for this purpose, the work on this product would be discontinued."

This specific product.

Q. Now, look at page 73336 and tell the Court what is reported there. Incidentally, what is the date of that page? A. That is October 1, 1951. The products which we had made previously were 100 per cent polymer and some were of coarse glasses and some were viscous liquids so we were wondering what would happen if they were in solution form, if we could get more useable mixtures, so we investigated the solubility of these adducts or reaction products that had been made previously in hexane, benzene and acetone.

Q. What was the result of that investigation? A. Well, this was just sort of a starter. None of the products were soluble in hexane. They were only partially soluble in benzene and some were soluble and some were

insoluble in acetone.

So we changed these materials which were originally either hard, glassy resins or viscous liquids to a more fluid state in some cases, but not in all cases. They are all detailed here on the page.

(546) Q. Look now at page 73337. A. Here we tried to make more dilute solutions of the polymers in benzene and we dissolved sufficient of the polymer of each of these different polymers in benzene to form a corresponding ten per cent solids solution and then these were spread out on a film and allowed to react with moisture and cure up.

Q. What were these products? A. These were being evaluated now as adhesives, specifically on that ABL project.

Q. Did you evaluate the properties of these adhesives? A. This page is concerned with curing them up and then it says, "See next page." There are some conclusions there. Number 1 and number 2 on page 38.

Q. What are the conclusions? A. Number 1 it says the adhesive layer reacts with atmospheric moisture or at least absorbs enough moisture during the brief interval preceding joining of the surfaces so that during the cure, that is after these two surfaces had been brought together, excessive gas evolution occurs. This creates many small bubbles in the adhesive layer, weakening the bond.

Observation number 2, the cured adhesive is excessively brittle. This prevents the distribution of strains (547) within the layer when stress is applied, making the bond susceptible to failure at the point of stress with consequent rupture of the entire bond.

A more elastic layer will reduce this effect.

Q. So that you were looking for a more elastic layer. A. Yes.

Q. Would you look, then, at page 73339, which is still in Exhibit Z. Tell the Court what it is reported that you did on that page. A. This is October 4th and 5th, 1951.

Essentially what we did here was to repeat some of the glycols that we had previously reacted with TDI and reacted them with MDI, which is a higher molecular weight, more polymeric type isocyanate.

Q. Why did you do this? A. Well, we were trying to get away from the brittle effect that occurred when we used TDI and, in fact, it says here that these molecules or these materials are more elastic than those made with TDI and should show more promise as adhesives.

Q. Were these the same types of diols that you first

used on September 4th? A. Yes.

Q. Now, I would like you to look at notebook pages (548) 73342, 73343, 73345 through 49, and then notebook pages 75851, 52 and 54, which are in Defendant's Exhibit AA, and tell the Court what is reported on those pages along with the dates. A. In the first place, we

would have preferred to make our polymers with TDI because it was cheaper than MDI, so even though our previous experiments had showed that MDI was superior, we still had a big interest in trying to make flexible materials from TDI.

So what we did was to go to higher molecular weight glycols. In other words, the ones we had tried so far, the hydroxyl groups, were fairly close together and now we were trying to get materials where they were further and further apart, like on those charts there, except these were diols.

So, for example, we tried polyethylene glycol, which we can abbreviate PEG from now on. PEG 600, carbowax 1000, which is a PEG 1000. Carbowax 1500, which is a PEG 1500, meaning 1500 molecular weight.

Mr. Kelton: May I just interrupt for a second, Mr. Heiss.

Your Honor, it would be much easier for us—I notice Mr. Heiss is turning pages, picking a page and then turning it—if he indicated where he was.

The Witness: Excuse me.

Q. These are the pages I read off. I was just trying (549) to move him along a little bit. A. We are talking now about 73342, 43. On those pages these pre-polymers were prepared with these aforementioned polyglycols and TDI or MDI.

Now, on 73345, which is dated October 15th and 16th, 1951, we evaluated these materials. We note here what their appearance was. For example, viscous amber liquid or a rubbery brown solid or a waxy yellow solid and so forth, as indicated on the pages.

These things were reacted with moisture, with water then to form foams, small samples of foam and these are also described on page 73345. Each foam is specifically described.

Then down below is the general comment, it says properties of foams can be tailored to various specifications by use of various polyols or isocyanates.

TDI with polyethylene glycols of about 600 molecular weight seem useful. It is expected that MDI will be best when used with lower molecular weight polyols.

Are we continuing now with 46 and 47; is that what we are doing?

Q. You just finished 45. A. I finished 45.

Q. Then look at page 73346. (550) A. Okay. On page 346 we now depart a little bit where previously we were reacting either TDI or MDI with one species of glycol, now we are mixing and we reacted TDI with a mixture of PEG 600 with tetraethylene glycol, which is really a low molecular weight PEG.

Q. What is the date of that page? A. That is October 17th to 18th, 1951.

So we made the pre-polymere, we reacted it with moisture to make a foam and we compared this foam to the previous foams and it says here, the second paragraph on that page, the foam is light colored, firm yet flexible, and has a greater volume and foams more readily than one of the previous ones, 73342B.

Then it says the following questions remain to be answered: Is a blend of glycols superior—well, is there any point in listing questions?

Q. You can list them; go ahead. A. Is a blend of glycols superior to the use of one glycol of intermediate molecular weight?

Would use of MDI and a polyglycol of lower average molecular weight produce superior foams to TDI and a polyglycol of higher average molecular weight?

Such an MDI product should be more hydrophobic since less EO links would be contained in the molecule. (551) We are already starting to notice that we need less hydrophilic tendencies.

3. Will the presence of excess TDI or MDI improve foaming action and result in superior foams?

Q. Now go to the next page, which I think is 73347.

What is the date of that? A. October 19, 1951.

Q. What is reported there? A. We are reacting TDI or MDI with different polyethylene glycols, PEG 400, PEG 200 and tetraethylene glycol. We are just making

the pre-polymers on this page.

Q. Then go on to the next page, which is 48. A. Here, instead of using, for example, PEG 400, we react TDI or MDI with a mixture of PEG 200 and PEG 600. As mentioned in this previous question, in other words, is there an advantage or not to using an intermediate molecular weight or to using a mixture of a high molecular weight and a low molecular weight material.

Then down below on the bottom of that page we go to a slightly different subject where we were starting to look at methods; although foams were interesting, we didn't always want foams. When we were making adhesive, we would prefer that it did not foam, so at the bottom of this page we are starting to look at another approach and what could we do to reduce the foaming of adhesives. This is how we branched out. (552) We were trying to find out what these things were useful for.

Some were foams and that was good, but we also wanted them not to foam in certain instances so we wanted to see could we get good foams and, number two, where we didn't want foams, what could we do to minimize the foaming reaction.

What was the next page!

Q. The next page is 73349. A. Well, this is more work where we were making the pre-polymers and emersing them into—no, wait a minute—here we were preparing the pre-polymer in solution form. No, I am still wrong. I am ahead of myself.

We took the previously prepared pre-polymers and dissolved them in benzene to make 50 per cent solutions, and then these were cast out and evaluated as adhesives. The results are tabulated at the bottom of that page.

- Q. Now, if you would, please, Mr. Heiss, look at Defendant's Exhibit AA. A. Yes.
- Q. And go, first, to notebook page 75851. What is the date of that page? A. October 31, 1951. were looking at-as I say, we noticed when we deposited these adhesive layers on a surface and allowed them to react with atmospheric moisture they would foam and we were trying to study the (553) foams and so we wanted the foams to be formed in a more uniform manner so we were investigating ways of making more uniform foams and the one that was investigated on this page was deliberately exposing the layer of adhesive, after it had been spread out, to water by immersing it into a water bath so that there was maximum possihility for contact with water. We just describe some of the results and some of the effects of having things dissolve in the water, which were of no really practical interest here with regard to either foams or adhesives. We were just feeling out way now to see what approcah we should take.
- Q. Would you look at the next page. What is the date there? A. November 1st to 4th, 1951.
- Q. What is reported on that page? A. We are noticing that some of these solutions that we made of these adhesives are starting to gell up now on standing around at room temperature, and we noticed that we had two types of solutions, some were uncatalyzed and some were catalyzed and that the catalyzed solutions gelled up faster than the ones that were not catalyzed.
- Q. Can you look, then, at page 75854. What is the date of that page? A. November 7, 1951. Here we are

again spreading the (554) adhesive layer out and deliberately immersing it in water or water containing various additives to see what types of foams we were getting.

- Q. Mr. Heiss, can you summarize or in summary describe the products that you had made thus far in this research program. A. Thus far we had reacted either TDI or MDI in excess with a variety of linear glycols of molecular weights from 31 up to 1500 so as to form NCO terminated pre-polymers or adducts. These adducts then had either been allowed to form a foam or had been evaluated as adhesives between two other materials.
- Q. Now, would you please go back to Defendant's Exhibit Z again and look at page 73341. What is the date of that page? A. October 5th to 10th, 1951.
- Q. What do you report there? A. Well, at the top it says, since higher molecular weight polyglycols seem to react with MDI to form stickier materials, further reactions to be made with some yet higher molecular weight materials.

So on this page we reacted MDI with PEG 600 and we made the pre-polymer, and when this was evaluated as an adhesive, it was spread in a thin film, it did not bubble nearly to the extent of some of the others. It says here it dries (555) to a rubbery film which adhere well to metal and glass.

Then it says, the use of polyglycols of higher molecular weight appears to approach the solution to this problem. It would seem that formation of high molecular weight triisocyantes would offer additional advantages. In view of the fact that long chains are desirable, ethylene oxide condensates with tri- and tetra and other polyols, such as triethanolamine, glycerine, pentaerythritol, et cetera, should form interesting—well, there is something wrong here. Anyway, it says "should form

interesting as adducts for di- or polyisocyantes." The "as" should not be in there.

Q. Is this the first disclosure, then, of your idea to use the EO condensates with glycerine? A. Yes. I was just trying to think—I haven't mentioned it up to now?

Q. No. A. Okay; right.

Q. The date of that? A. October 5th—well, this specific paragraph is dated October 10, 1951.

Q. Then would you look at page 73344. What is the date of that page? A. October 12, 1951.

(556) Q. What is recorded there? A. It says ABL Project. Preparation of high molecular weight triisocyanate by reaction of TDI or MDI with glycerine EO condensate.

Then there is an asterisk and it defines this glycerine EO condensate as one mol glycerine plus 10 mols EO prepared by Hector Flores, December 20, 1950, no notebook number, unfortunately, for the condensate.

Q. This is the condensate, the EO condensate that you testified that you got from the detergent group? A.

Right, the one which they had made previously.

Q. They had made it previously for use in detergent work? A. Right.

Q. What were your observations of the reaction on this page, Mr. Heiss? A. We didn't like this reaction. It got out of hand, was uncontrollable and we make the note here that instantaneous reaction forming hard infusible product. Needs more moderate procedure.

Then below it says, October 15, there is a note: it is probable that the above reaction can be run smoothly

in solvent media.

Q. Did you do further work on the materials that you (557) describe on page 73344 to convert them into adhesives? A. No, we didn't do any more with those.

Q. Why was that? A. Well, because the reaction hadn't gone properly and it just didn't seem right, so

we didn't waste any more time on those particular examples.

Q. Throughout this period, though, you were using diols in all of your reactions, weren't you? A. Yes.

Q. Why were you using the diols instead of the ethylene oxide condensate that you said you had gotten? A. Well, okay, I see. This particular material was in limited supply. It was a thing that they had made experimentally.

Q. You mean the EO condensate? A. Yes, of glycerine and even later on the ones that he made for us, the PO condensates, these were one of a kind items which

we had on hand in limited supply.

On the other hand, all these glycols, like the PEG 200, 400, 600, we could go out and buy 5 gallons of those on the market, so we had a lot of problems here with trying to control reactivity and the effect of impurities in the reactants and that type of thing, so rather than waste our precious research type material of endeavor, we used up the (558) commercial linear materials.

Q. If you will, then, look at page 75858, which is in Defendant's Exhibit AA. A. Yes, there are just some comments here, no experimental work.

Q. What is disclosed on that page? A. This is dated November 14, 1951, and we say here just some general

comments on our experiences to date.

Q. These are your experiences? A. Yes, right, to my experiences to date.

Due to the similarity between these materials and the German diisocyanate polyester adducts, as used in Vulkollan; for example, it might be wise to consider their uses in broader scope. This would appear to offer three advantages at least.

1. Easier circumvention of the German Vulkollan patents, which were based on polyesters.

2. The ether linkages of the polyglycol adducts should be more stable than the ester linkages of the polyesters. These latter seem to be less resistant than the urethane linkages in the Vulkollan's and so elimination of these weaker bonds should improve overall product stability.

Comment number 3. The use of polyglycols or other ethylene or propylene oxide condensates offers unlimited (559) variety in building up molecules of various degrees of linearity or non-linearity.

Q. Is this the first time that you have mentioned your idea of using propylene oxide condensates? A. I believe so, yes.

Q. And the date of that page? A. November 14, 1951.

Q. Now turn to page 75863. A. Yes.

Q. Would you tell the Court what you reported on that page. A. That is dated November 20th to 23rd, 1951.

Now we are back on the ABL Project. We are reacting TDI with the glycerine EO condensates that gave us so much trouble before. We are now trying to run the reaction in a solvent, in acetone, anhydrous acetone. We didn't prepare the adduct first and then dissolve it. Now we are trying to run the reaction right in the presence of the solvent and hope to moderate this uncontrollable reaction that we had the first time.

Let's see if we did or not.

Q. What was the result of that? A. Here is part of the comments. It tells how the reaction was run and we are proceeding along and then it (560) says: After about half of the glycerine EO condensate has been added, 15 minutes, the liquid suddenly becomes viscous and turns into a crumbly soft solid. Addition of remaining glycerine EO was terminated and the remaining acetone added.

The solid does not all go back in a solution and some more comments, but, in other words, this reaction, even

in the presence of the solids, still did not react properly because the material should not have cross-linked up to that extent.

Q. Did you evaluate this material at this time? A. The material was insoluble; we couldn't; even after we put it in hot solvent it would not dissolve, so there is a comment here; This approach abandoned.

Now there is another run on this page. Let's see what is different.

Oh, instead of using acetone as the solvent we used anhydrous dioxane, which is a better solvent generally. Now we were at least able to add all the glycerine EO, we were able to get all the reactants in. The mixture is a viscous opaque yellow material. In other words, it wasn't really a solution, but a liquid dispersion.

Then on November 23rd we distilled off the dioxane under reduced pressure. The product still didn't look right. It wasn't uniform. It was a dispersion of high melting insoluble (561) material in another phase, so we still didn't like it.

Down below it says this type adduct is too insoluble to be of much use. However, it may be possible to blend it with a polyglycolisocyanate adduct or to react isocyanate with a mixture of polyglycol and glycerine EO.

Q. You have talked in your testimony today, Mr. Heiss, about pre-polymers in some of these areas. I think it would be wise if you would explain what a pre-polymer is. A. Sometimes in the testimony here we will be calling them pre-polymers and other times we call them adducts, but in essence it means taking a thing, such as a polyethylene glycol, which is terminated in hydroxyl groups, and reacting it with an excess of a di-isocyanate so that you convert it to a material of higher molecular weight that is now terminated in NCO groups and is therefore—could therefore be considered a high molecular weight polyisocyanate.

Q. Would a pre-polymer further react? A. Yes.

Q. You can react it with something else. A. You can react it with a variety of things. You can react it with anything that isocyanate will react with.

Q. Did you know what pre-polymers were at the time

you were working in 1951? A. Yes.

(562) Q. Now, look at page 75864, which is still in Defendant's Exhibit AA. A. That is dated November 23rd to 27th, 1951, and now in response to the comments made previously, we are trying to modify this reaction and react MDI not with the pure glycerine EO adduct, but with a mixture of the glycerine EO adduct with PEG 400.

The first time we tried it we again got an uncontrollable reaction in spite of the fact that the glycerine EO material had been diluted down with the PEG.

Then we tried it again and we got a little more success. We were able to get a very viscous prepolymer which we could convert into a foam and the comment is made here that foams and films made from this adduct seemed to be more flexible than those made from 75860C, the previous material.

Q. Was this an evaluation then of the material that you made? A. Right, the preliminary one and the com-

ment is made, more tests will have to be made.

Q. What does it mean when you say that the foam is more flexible and harder? A. Well, maybe that is a contradiction in terms, but what I meant was it was firm, but more resilient. A foam that is harder is stiffer. A stiff foam can or cannot be (563) resilient. In other words, if you push on a foam and deform it, it can either recover in a rapid fashion or a slow fashion. If it recovers in a rapid fashion, you say it is resilient.

Q. Would that he something like the difference between a hard mattress and a soft mattress? A. Right.

Q. Was the foam that you made as you report on page 75864 an improvement over the other foams that you had made? A. Well, on November 27th, after the foam had had a chance to sit at room temperature for a day, the comment is made that the foams made from this adduct appear to be harder on aging so that after 48 hours they are harder than those made from 75860C. That is all it says on that page. I was going back to see what 60C was. 60C, the foam that it refers to, was made from MDI and only polyethyleneglycol 400. In other words, there was no branching in it.

Q. Did you feel that the foam that you had made on page 75864 was an improvement therefore? A. Yes.

Q. Look at page 75881 in Defendant's Exhibit AA. A. That is dated January 15th to 17th, 1952, and here we were trying something else. We were making adducts, but instead of trying to make an NCO terminated adduct, we were reacting an excess of the polyol with the isocyanate to form (564) OH terminated reaction product.

Then we had hoped to mix this with an NCO terminated adduct and therefore get a controlled reaction of the OH and NCO groups, but this page really describes the preparation of the OH terminated adducts.

Q. What were your observations about this particular adduct? A. Well, the main thing was that due to the terminal OH groups, this material was water soluble and hydroscopic, which was rather surprising to me.

Q. This was an EO adduct material? A. Right, terminated in OH groups.

Q. Now, if you would look at page 75883 and tell us what you did. A. Well, this is where we were actually mixing these adducts prepared on 7/—75881 with some other NCO terminated adducts to see what type of products would result.

Q. What happened in this reaction? A. Some of them foamed.

Q. This is now still an EO adduct. A. Right. I suppose the general comment that is of interest here is the last sentence on that page where it says, talking about which ones had foam and what types of foams were formed, it says all the others had foam: The firmness of the (565) foam decreasing as the percent of 75881B increased.

Now, 75881B was the adduct of TDI with glycerine EO condensation product, but OH terminated.

Q. What were the implications of this as far as you were concerned? A. Well, there again we seem to show that putting branching in in a different way, but still in a high molecular weight form, now we were using it as a curing agent for the NCO terminated prepolymer, the more branching you put in the firmer the foam.

Q. Now would you look at page 75584 and tell us what

you report there.

Page 75584. A. Well, this is where we became suspicious of why we were having trouble reacting the glycerine EO condensate isocyanate and rather than read all this, I will just summarize and say that we came to the conclusion that the difficulty lie in the fact that when EO or PO are condensed with glycerine or other materials, an alkaline catalyst is used. If this alkaline catalyst is not neutralized prior to the isocyanate reaction, you get an uncontrolled reaction with the isocyanate.

Since the polyethylene glycol products, which we could huy on the market, were commercial items and were presumably more carefully neutralized and purified than our (566) experimental products, we didn't have that much trouble with them, although later on as we became more sophisticated and were more sensitive to impurities we could see that even there we had to be careful.

But in essence these experimental products that we got, the catalyst was not properly neutralized. The materials, the condensates of EO and PO were alkylene and therefore when we tried to react these with isocyanate we got uncontrollable and non-reproducible products, reactions of the products.

Q. All right, Mr. Heiss, if you will look now at Defendant's Exhibit AB and pages 79054 and 79055.

Wait, before you do that, would you mind putting in the record the date of page 75584, please.

The Court: The number of the page is 75884; is that the page you are talking about?

The Witness: Yes.

(567) The Witness: That is dated January 17th and it should be 1952.

Q. Now look at pages 79054 and 79055. A. Both these pages are dated February 20th to 27th, 1952.

Q. What do they report? A. The title is Effect of Increased Functional Groups, and here we are continuing the approach of reacting an excess of TDI with a mixture—no, that is not right. Wait a minute.

Rather than react them in mixture form, what we are doing here is we are making an adduct from an excess of TDI and a linear polyglycol, such as PEG 400. We are separately making an adduct of an excess of TDI with a branched higher molecular weight polyol and then we were considering whether we should mix the two together.

Q. Did you get useful products on that page? A. These samples were all prepared as 50 per cent solutions (568) in anhydrous benzene and they contain catalyst—no, I am wrong about this. We did not separately prepare the adducts, excuse me. We mixed the

PEG 400 with an appropriate amount of these branched materials and then we reacted that with an excess of TDI in the solution to get the adhesive solution and some of the materials we used here were pure glycerine, glycerine plus 10 EO, glycerine plus 15 EO.

Now here is a note number 1: We are getting some more adducts apparently because it says here the glycerine plus 10 EO and the glycerine plus 15 EO prepared by Milton Kosmin at Central Research. That was another Monsanto Laboratory at Dayton. Per, apparently, a memo from W. B. Satkowski to M. Kosmin, dated 10-17-51.

So I guess we were getting help from the Dayton Labs on making some of these adducts.

Q. This was an EO? A. An EO condensate, right.

Q. Who was Mr. Satkowski? A. Mr. Satkowski was the gentleman we mentioned before who was the group leader in charge, as I recall, of the detergents laboratory, which was responsible for making surfactants based on ethylene oxide.

Q. Who was Mr. Kosmin? A. Mr. Kosmin was working—well, among other things, (569) he worked on EO condensates at the Daylon Laboratory, but that was the Central Research Lab. They weren't concerned with any specific products.

Q. Now look at page 79055, which is the next page, and tell us what you were doing there. A. Now we are describing—on the first page we wrote down the weights we were going to use to make these compositions, and on page 55 we are continuing with what actually happened.

Now here, where we tried to use these EO condensates which we had gotten from Kosmin, we had the same old problem again, uncontrollable reactions, and the comment is made here, reactions GHJ and K, which are the ones involving these EO condensates, were very violent,

blowing the contents out of the bottle. These were repeated using half as much of the polyol Ks and a greater amount of PEG 400.

In other words, we were diluting down the trouble-some material.

These reactions which we put an R in front of the letter for Revised, they were G, H, J, K with an R in front of them, were less violent than the above mentioned reactions, but were still more vigorous than normal.

So here again we make the comment, it is possible that the catalyst used in the preparation of these EO condensates (570) is also catalyzing the TDI reaction. Efforts will be made to neutralize these EO condensates before drying them. Also, perhaps acetone would be a better solvent than benzene.

- Q. Now, if you look at the next page, 79060,— A. Dated March 4, 1952.
- Q. What do you report on that page? A. Well, by this time we had accumulated a lot of experimental data and these things looked very intriguing. We hadn't done anything patent-wise and it still didn't seem worth-while to put in a disclosure because we were still generating information, so the way we get around that to preserve a date was we would write what we called an idea sheet in the notebook and then we took special pains to make sure that such a page was witnessed and signed by at least two other people and that is what this page is, and it is entitled, Reaction Products of Isocyanate and Polyhydroxy Compounds And/Or Their Ethylene or Propylene Oxide Condensates.

There is a lot of writing on that page where we summarize our past experiences.

Q. Who witnessed that page? A. Paul G. Geminhardt and William B. Bennett, and they witnessed this on March 4th, 1952.

Q. Were they Monsanto employees? A. Yes, they were Monsanto employees.

(571) Q. You have mentioned here on your idea sheet, as you call it, which is 79060, and you again mention the use of propylene oxide condensates. A. Yes.

Q. Had you tried them yet? A. March 4th, 1952, I

don't believe we had tried them yet, no.

Q. Do you know why you hadn't tried them yet? A. They had not yet materialized from Mr. Satkowski's group.

Q. Now, would you turn again to page 79061. A. Another idea sheet dated March 4, 1952 where we are concerned with other types of hydophobic polyols and these were—well, the title here is Unsaturated Reaction Products of Isocyanates and Polyhydroxy Compounds And/Or Their Ethylene or Propylene Oxide Condensates.

Again this was witnessed by Geminhardt and Bennett

on the same date.

Q. Will you look now at page 79062 and 79063. A. Well, these are dated March 5th and 6th, 1952, and now we are really starting to examine this problem of why these EO condensates were so uncontrollable and we are looking at this business of the suspicion of ours that the alkylene, unneutralized alkaline catalyst was causing the trouble, so (572) these merely record a bunch of examples where we reacted the EO condensates with isocyanate as received and after we had neutralized them with HCL and we discovered that the ones which were neutralized with HCL gave much nicer reactions and so our speculations had been correct. The alkylene impurities were causing the trouble.

Q. Now can you look at page 79078 and 79083 and tell us what is reported on those pages. A. 79083 is dated March 31 and April 1, 1952. Now, at the top of the page it explains what we are doing. It says: Due to the fact that the prepared foams tend to soften—now

these are the foams made from these EO condensates—it will be interesting to determine the effect of increasing the cross-linking during curing. This will be in addition to increased rigidity by decreasing molecular weight of the glycol. Cross-linking should provide a different sort of rigidity.

This again is related to some previous work where we are—just a second now—well, I have to go back to 79074. On that page we made some NCO terminated prepolymers and we converted them to foams by reacting this prepolymer with a mixture of water, a catalyst, which was N-methylmorpholine and with the water we replaced part of the water with ethylene glycol. In other words, here we had the option of (573) curing up the adduct with only water or with a mixture of water and ethylene glycol.

Now, the ethylene glycol could cure up the isocyanate prepolymer without forming gas and would form a ure-thane group, so we were examining really getting foams of different density because we were decreasing the water content.

To get back to page 79078 instead of adding water, ethylene glycol to the water, we were adding other materials such as ethylene glycol glycerine and the glycerine plus 10 EO adduct, the glycerine plus 15 EO adduct and so on.

Apparently we were not using the neutralized EE adducts on this page because there is a note here, it says, these reacted—now, wait a minute—no, that is something else; excuse me, delete that.

All these foams softened on standing. In order of decreasing firmness they are B and E, A, C and F, D and G. All samples suffered more shrinkage than A.

Q. What did this mean to you? A. The comment at the bottom of the page is: Increasing cross-linking during the foaming reaction does not have much effect on

form rigidity, contrary to what we saw before where we built the branching into the prepolymer.

- Q. Now would you look at page 75858, which is in Defendant's Exhibit AA. (574) A. 75858.
- Q. What is the date of that page? A. This is the one we read before.
 - Q. That's right. A. November 14, 1951.
- Q. This, I believe you testified, is the first time you wrote down the idea of using the propylene oxide condensate. A. Yes.
 - Q. Is that page in your own handwriting? A. Yes.
 - Q. Did you prepare it? A. The page?
 - Q. You filled out that page yourself? A. Yes, right.
- Q. Whose idea was it to try these propylene oxide condensates with diisocyanate? A. Mine.
- Q. I think that you have indicated that you asked Mr. Satkowski for them, but would you explain a little more fully how you went about getting them? A. Well making these condensates is kind of an elaborate process. You need certain types of equipment. The detergent group was set up to react ethylene oxide with these various polyols and when I talked to Satkowski he said he felt that (575) when they had a chance they could modify their equipment slightly and get in some propylene oxide and when they had the time available they could then make these propylene oxide condensates for us, so I merely put in the request to him that as soon as convenient he make them for us and while we were waiting we turned our efforts to other work.
 - Q. You ultimately did get the material? A. Yes.
- Q. I think you have already testified that Dr. Flores made it. A. Yes.

(576) Q. Mr. Heiss, would you look at Defendant's Exhibit AF for identification and tell the Court what is that? A. Exhibit AF is one of the original bottles made

by Hector Flores on October 5, 1952, his notebook number 84732. It is glycerine plus 9 propylene oxide.

Q. Does that bottle contain some of the glycerine propylene oxide condensate that you received? A. Yes, this is some of the original material in the original bottle.

Q. Now, do the same with Defendant's Exhibit AG. A. Sample AG is another material received on the same date from Hector Flores, his notebook number 84729. It is glycerine plus 6.5, I think, PO.

There are two other samples in that series. He made 3, 6, 9 and 12. I found one more in the lab just before I came, but I didn't bring it, and I don't know where the other one is. These are part of a series of four.

(578) HECTOR FLORES, called as a witness by the defendant, and being first duly sworn, testified as follows:

Direct Examination by Mr. Sweeney:

Q. Where do you reside, Dr. Flores? A. Mexico (ity.

Q. Would you please give us a brief summary of your educational background? A. I finished BS in chemistry in 1939, University of Puerto Rico. I worked for a couple of years and went to the graduate school where I finished an MS at the University of (579) Florida, 1942.

I started work for the doctor's degree and then went into the army, returned after the service and in 1947 finished a PhD at the University of Florida.

Q. What is your present employment? Where are you presently employed? A. In Mexico City.

Q. By whom? A. Searle of Chicago, a subsidiary in Mexico.

Q. What is your position with Searle in Mexico? A. Vice president of the operation, general manager of the chemical division.

- Q. Did you work for Monsanto Chemical Company? A. Yes.
- Q. Can you tell us the dates and the time you worked for Monsanto? A. From January 1949, to January 1953; exactly four years.
 - Q. Where were you located? A. Anniston, Alabama.
- Q. What was your position with Monsanto? A. I was a research chemist.
- Q. What duties did you have as a research chemist? A. Most of my work there consisted of synthesis of (580) products in different areas, in the antioxidant field, in the detergent field, in the—similar things, phosphates.
- Q. And you carried out condensation reactions doing this? A. Yes, I did.

Mr. Sweeney: Will you mark this notebook as Defendant's Exhibit AH?

Your Honor, there are only four pages of this hook that have anything to do with this matter, so that I have only copied the four pages.

(Defendant's Exhibit AH marked for identification.)

- Q. Dr. Flores, I hand you a notebook which has been marked as Defendant's Exhibit AH and I ask you if you can identify it. A. Yes. This is my handwriting and it's one of the notebooks that I filled while at Monsanto.
- Q. Were the entries in the notebook, Defendant's Exhibit AH, made by you while you were performing work? A. Yes.
 - Q. I'll refer you to page 84729 of that notebook. What is the date of that page? A. August 4, 1952.
- Q. And can you tell the Court what work is reported on that page? (581) A. Here is described the work where one mol of glycerin was reacted with 6.6 mols of PO, propylene oxide. I give here the details of temperatures

and pressures and the way I purified the material at the end which was by, I read from there, added 100 cc of water, blue ink carbon dioxide for ten minutes, heated at 110 degrees centigrade for the vacuum for two hours, filtered and labeled it glycerin plus 6.6 PO.

- Q. What catalyst did you use? A. Potassium hydroxide
 - Q. I ask you to look at Defendant's Exhibit AG.

Is that the condensate that you report preparing on page 84729 of your notebook? A. It appears to be the material that I prepared at that time.

Q. Is that the bottle that you put the material in? A. It looks very much like.

Q. And is the label that's on that bottle the label that you prepared? A. Yes. I used to type them myself and it looks to me like my typewriter.

Q. Now, if you will, look at page 84732. What is the date of that page? A. August 4, 1952.

(582) Q. What do you report on that page? A. This is the reaction, the condensation of 9 mols of P() with one of glycerin. Here I reacted 138 grams of glycerin which is 1.5 mols, with potassium hydroxide as catalyst with 780 grams of propylene oxide.

I give here the temperatures and pressures and times and the purification of the material.

Q. If you will look at the other bottle, which is marked Defendant's Exhibit AF, I'll ask you if that is the condensate that you report you prepared on page 84732? A. It looks very much like what I prepared.

Q. Is that the bottle that you put the condensate in? A. Yes; it appears to be.

Q. Look at the label. Is that the label that you prepared? A. Yes, it is.

Q. If you will look at 84735, what is the date of that, doctor? A. August 6, 1952.

- Q. What do you report on that page? A. The description of the reaction of glycerin plus 13.2 mols of propylene oxide. Here I again give the times, the temperatures, pressures and the results of this preparation. At the end I also describe the purification and how (583) it was labeled.
- Q. What was the catalyst used here? A. Potassium hydroxide, the same as before, and the same proportions as the other preparations.
- Q. Look at page 84728. What's the date of that page? A. August 1, 1952.
- Q. What's reported there? A. The reaction of glycerine plus 3.1 PO. Again I mention here the quantities involved, which was 368 grams of glycerine, 8 grams of potassium hydroxide and 696 grams of PO. I give here the times, the pressures, the temperatures and how it was purified and how it was labeled.
- Q. What was the purpose of your preparing the four condensates that you have just testified about? A. I don't know the ultimate purpose but I was asked to prepare them.
- Q. Who asked you to prepare them? A. My immediate supervisor, Mr. Satkowski.
- Q. What did you do with the four bottles of condensate after you prepared them? A. Turned them over to Mr. Satkowski.
- Q. Do you recall about when you were asked to make these condensates by Mr. Satkowski? A. Several months before T prepared them.
- (584) Q. Why is it that you didn't prepare them earlier than August of 1952? A. There were several things. Number 1, I was working on several projects interesting to me, exploratory work, involving also PO, the reactions with certain alcohols and PO and then with EO, which I was interested in.

Second, I considered that this reaction should be carried out with anhydrous glycerine and the material received,

analyzed, I remember, a high content of water and I did not want to start with that. When we got dry material, we reacted it, the two things combined—absence of a reagent and other interests that I had.

Mr. Sweeney: I have no other questions of this witness.

The Court: Mr. Kelton, Mr. Blecker?

Mr. Sweeney: I suppose I should offer Defendant's Exhibit AH into evidence, which is the notebook pages.

The Court: You are offering pages 83728, 29, 32 and 35.

Mr. Sweeney: That's right, your Honor. I only need the four pages. The rest of the notebook has nothing to do with this project.

The Court: All right.

Mr. Kelton: No objection.

(585) The Court: Received.

(Defendant's Exhibit AH received in evidence.)
Mr. Blecker: We would just like a minute to look at the rest of the notebook.

The Court: Of course.

Cross Examination by Mr. Kelton:

Q. Mr. Flores, will you turn to page \$4728 of Exhibit AH.

Was it common for you to carry out operations in a three-necked flask? A. Yes, sir.

Q. With regard to page 84729, this appears to have been carried out in a two liter—what you described as a two-liter flask, is that right? A. Yes, sir.

Q. Had it been common for you up to that time to carry out reactions in a two-liter flask? A. Yes.

Q. Prior to August 1st of 1952, had you carried out or made condensates employing ethylene oxide? A. Yes, sir.

- Q. Had you made condensates of ethylene oxide and glycerine? (586) A. Yes, sir.
- Q. Had you, prior to August 1, 1952, employed propylene oxide? A. Yes.
- Q. With regard to the experiments on page 84728, 84729 and 84732 of Defendant's Exhibit AH, what, if anything, was different from your previous production of condensates of ethylene oxide and glycerine, except that you used propylene oxide? A. This was the first time I was using anhydrous glycerine with propylene oxide and the condensation was between two liquids.

Other reactions of glycerine and ethylene oxide was with normal industrial grade glycerine, which was not nonhydrous and the reaction was a gas, ethylene oxide and the glycerine liquid. That's the only difference.

- Q. Do you mean that in the case of preparing ethylene oxide—glycerine condensates, you were unsuccessful, had been previously unsuccessful in carrying them out or had you had success? A. With glycerine?
- Q. Glycerine and ethylene oxide. A. Most likely, and that's a speculation, there were mixtures of condensations of ethylene oxide and glycerine and (587) polymerization of ethylene oxide.
- Q. In other words, it wasn't a condensate, are you telling me that? A. Yes, but not a pure, 100 per cent.
- Q. When were you asked to prepare a glycerine propylene oxide condensate? A. I cannot precisely remember the date exactly but it was some time in 1952. Could have been March or April but I don't have any recollection.
- Q. Didn't it occur to you then to use the propylene oxide in the same way that you had used the ethylene oxide with glycerine to secure a condensate? A. In those days I had run many, many ethylene oxide condensations, hundreds, with acids, with alcohols, different things, and once you run a reaction many times you are confident and more or less know to what extent you can go.

On propylene oxide, the experience was limited. I had run it only in long chain alcohols.

Q. But the equipment that you used with the propylene oxide condensate was the same as you had used with the ethylene oxide? A. No. It was different.

Q. How was it different? A. With ethylene oxide, we were passing the gas into (588) the top of the atmosphere and, during the stirring, it was absorbed and reacted.

For the propylene oxide, we devised a dropping funnel, an additional funnel, where we pressurized it with the same pressure in the reaction and dropped the liquid drop by drop into an excess of the alcohol involved.

Q. With a propylene oxide and an alcohol, will you accept that glycerine is an alcohol, Mr. Flores? A. Yes.

Q. And an alcohol, made with glycerine, how did you do that? A. Placed the glycerine in a flask which had three necks. To the glycerine, added the catalyst, which was potassium hydroxide; purged the system with an inert gas, such as nitrogen, and heated it up to a certain temperature.

In those days we were carrying all those condensations in the vicinity of 150 degrees Centigrade.

Once we had attained that temperature and the system was under a blanket of nitrogen, then added slowly, drop by drop, the propylene oxide and, if the pressure went up, that meant that the reaction was not taking place and had this volatile material and created pressure.

That was our method to know how fast to add the propylene oxide.

(589) Q. Is that the method that you used when you worked with propylene oxide? A. Yes, sir.

Q. And that is the work that you had done before.

For example, combining capryl alcohol with propylene oxide on July 28, 1952? A. Yes, sir.

O. Mr. Flores, did you make any analysis of the product that you made back in August of 1952, August 5, 1952, namely, I think probably illustrated by Defendant's

Exhibits AF and AG? A. In the sense of a direct physical analysis, no, but we determined the pH as indicated in there and, in addition, heated the material on the vacuum for a certain amount of time and it did not lose any weight, indicating there was no propylene oxide present. Everything had been condensed.

The Court: What did you say? You determined the pH?

The Witness: The pH is a determination of acidity or alkalinity.

- Q. Let me ask you, in view of your last comment, Mr. Flores, do you recall how long it took you to make the preparation for illustration on page 84729? A. One day's work.
- Q. Didn't you make any attempt to purify your product? (590) A. No. The only purification was to neutralize the catalyst, then evaporate the moisture hat was—the water was added before the neutralization, so it would make it simpler and faster and, then, the water was evaporated and the material was filtered.

Once the water was evaporated and the salt formed, in this case, potassium carbonate, settled out and filtered, that was the purification.

Q. If it took you only one day, you could have found the time in the preceding period, if you had been asked to, couldn't you? A. My recollection is that I was asked once and earlier in the year and let it ride.

Then I have a very distinct recollection that one day Mr. Heiss came around to talk to my boss and the same week I started working on it. I was more or less pressed but it must have been July, because I worked in August.

Q. It's your testimony that Mr. Heiss talked to your hoss. Mr. Satkowski, about a week hefore you— A. Could have been a week.

Mr. Kelton: That's all, your Honor.

The Court: Mr. Sweeney.

Re-direct Examination by Mr. Sweeney:

- (591) Q. The occasion that you talked about when Mr. Heiss came and talked to Mr. Satkowski, was that the second time you had been asked to make the material? A. The second time.
- Q. You were asked earlier in the year? A. But I cannot say the precise time.

Mr. Sweeney: No further redirect, your Honor.

Re-cross Examination by Mr. Kelton:

Q. I am curious about one thing, Dr. Flores. The heading on these pages, 84729, 847—

Mr. Sweeney: Your Honor, this is clearly beyond the scope of my redirect. I only asked him about— The Court: Let me hear the question, first.

Q. In each case, it seems to me to be alkylene oxide condensates.

Now, that was applicable to propylene oxide. Was it in your view applicable to ethylene oxide? A. Yes, both are included in that group.

(592) HERBERT L. HEISS, resumed the stand.

Direct Examination Continued by Mr. Sweeney:

Q. Mr. Heiss, just to put you back in perspective, when you got off the stand you had just identified the condensates which are Defendant's Exhibits AF and AG.

Did you actually use materials from those two bottles to make your things? A. Yes.

Q. Will you look at page \$4531 of Defendant's Exhibit AC. Do you have it? A. Yes. September 19, 1952.

(593) Q. Will you tell us what work you did as reported on that page? A. Well, now we are jumping back to

another series of problems we had.

You notice this page is entitled "Special samples for sales," and, as I mentioned, we had these research projects. One was working on foams, one was working on the deactivity problem of neutralizing the catalyst and this was in conjunction with the adhesives and, on this page, we were reacting, in henzene solution, to form a 50 per cent solution TDI with one of the glycerine PO's received from Dr. Flores, and the one we used was his notebook 84729, which was the glycerine plus 6.6 PO.

Q. Mr. Heiss, would you mind repeating the testimony about this page. A. This was one of the aspects of our work where we were working on adhesives and on this page we were making a special sample for sales to be evaluated as an adhesive and what we did on this page was to react TDI with one of the materials we had just received from Hector Flores, namely, his notebook number 84729, which was glycerine plus 6.6 PO. And this reaction was performed based on our previous work now, formed adhesive in solution form.

In other words, the benzene was present at the (594) time of reaction so that we, the resulting product, was in the form of a solution, a 50 per cent solution in benzene.

Now, the pertinent comment here is about halfway down the page, where it says September 22nd, which was a few days after we made this, on standing over the weekend, this product changed to a clear transparent solid containing many fissures. This looks like a good way to solidify Aroclor.

Q. Is the data on page 84531 in your own handwriting, Mr. Heiss? A. Yes, and then I should mention that further down below we repeat the experiment where we

replaced the benzene with Aroclor 1242, which was one of a series of many Aroclors, to see what would happen.

Q. Did you actually fill that page out? A. Yes.

Q. Was that work done under your supervision? A. I did this work myself at this page, I am pretty sure I did. Otherwise, it would have been in Billy Davis' notebook.

Q. Was this the first product that you made by reacting propylene oxide based triol with polyisocyanate? A. I believe so. To be absolutely sure, I should look at all the previous notebook pages but since we have (595) done this before, we came up with this number, I presume this is the first time we used it.

Q. You mentioned Aroclor. Would you explain what that is? A. Aroclor is a Monsanto trade name for a series of materials which were either biphenyls or terphenyls, which were chlorinated and these materials were generally used for the most part as insulating oils in transformers. They had good electric properties and they were nonflammable. They were used also for other purposes but this was the main reason for their existence as a commercial product.

Q. Would that be the dielectric, then? A. Yes.

Q. Could they also be used for potting compounds? A. Not as long as they were still liquids. Potting compound is a material used where an electronic assembly is put together, assembled and soldered and dipped into a fluid and then the fluid is caused to polymerize, so that when you are through you have this electron assembly protected and scaled from moisture in a resin which has to be a good insulator.

We reasoned, since the Aroclor we knew had good insulation properties, and since they were nonflammable, (596) and since we were now able to solidify them, we thought this might be a good approach as a means of making an electrical potting compound.

That's why we got interested now in deliberately causing these Aroclor solutions to gel up or solidify.

Q. In other words, you just said that you thought the reaction that you ran on 84531 would be good for gelling Aroclors? A. Yes.

Q. Can you explain what you meant by that? A. Well, we accidentally noticed that it gelled up our adhesive solutions, which we didn't want, so we didn't like that part of it but we also had interest always in other Monsanto products, so we knew that the benzene was just there as a solvent and so we reasoned that if we could gel up benzene, we could probably gel up other fluids, also.

They didn't have to be low boiling solvents and the one we chose was Aroclor, because we were interested in making a potting resin out of it.

- Q. Is Aroclor a high boiling liquid? A. Yes, very high boiling.
- Q. Did you run reactions of propylene oxide condensates in Aroclor? (597) A. Yes.
- Q. What role did the Aroclor play in this operation? A. It didn't play any role in the reaction itself. The Aroclor was merely something which was there and which was caused to solidify because of this urethane polymer which was forming in it in solution.
- Q. The Aroclor was a solvent? A. Sir, it could be considered a solvent, technically, except it was not volatile, so it didn't evaporate.

For example, this benzene solution that gelled up, if we took it out of the bottle and left it open to the air, the benzene would evaporate, the resin would try to shrink and crack. If we gelled up an Aroclor solution which would not evaporate, it remained as a tough resin which was a good electrical insulator and which would not burn.

Q. The Aroclor played no role in the reaction at all? A. No. It was like making jello. You dissolve a thing in water and it sets up, that type of thing.

Q. Will you look at page 84522 of notebook Defendant's Exhibit AC.

I am also interested in having you look at 84526, so that you can consider them more or less together. (598) A. Well, these even refer back to other pages, 84520, but previous to the time that we had received this condensate from Hector, we had discovered that we could gel up Aroclor by reacting isocynate with various components while they were dissolved in the Aroclor and that was done on, for example, 84522, and some of that was summarized on 84526.

On those pages, the material that we used to react with the TDI was TEA, triethanolamine.

Q. What's the date of those pages, Mr. Heiss? A. 522 is dated August 13th to 18th, 1952, and 84526 we have a summary which we sort of considered an idea sheet and went to the trouble of having it signed and witnessed and it's dated August 20, 1952.

In other words, I was going to say, we were capable of solidifying Aroclors and that's what we were working on about this time when Hector's PO condensates became available to us so, since this is the project we were working on, that's the first place they were tried out, in these Aroclor products.

Q. Do you say anything about the Aroclor being a solvent on those pages? A. Well, yes. Maybe by implication, but here is what I will read in the first paragraph on S4526:

(599) "Past work on these materials has indicated that the Aroclor probably does not enter into a chemical reaction with the TDI or TEA but is present as a dissolved inert ingredient in some sort of very high tarred, high melting polymer formed by the TDI and TEA."

In other words, there we are saying it's not a reactant but an inert material.

Down about the middle of the page, "It has been shown that Aroclor 'solutions' of various TDI—" and then it goes on, but there is the word you wanted, I guess, "solutions".

Is there any point to reading the rest of it?

Q. I'm not sure. Perhaps you might just complete the record. A. "Reaction products can be considered as types of thermoplastic (reversible) or thermosetting (irreversible) resins according to the ingredients used in the method of reaction. The reason that these materials may be considered unique from the standpoint of formation is that the thermoplastic or thermosetting resin is prepared in a dispersed form where-I should really have said whereas-but I said where it is very unlikely, for instance, that TDI-TEA polymer could ever be formed in a pure state and then combined with an equal weight of Aroclor to form a useful (600) resin. This is easily done by heating TDI and TEA in the presence of Aroclor. Apparently, the polymer is formed in an extended lattice that securely retains the Aroclor within itself. furthermore quite probable that materials other than Aroclor or halowax will be operable—continued to the next page—use of other nonvolatile liquids would result in the formation of resins that could be used as casting or potting materials in various electrical or mechanical applications. Proper choice of starting materials (isocyanates, active hydrogen compounds, inert liquids) would result in such qualities as good electrical properties, good fire resistants, good optical properties, good physical properties, etc.

"Use of more volatile liquids could result in the formation of solid fuels (where the liquid is flammable) or might produce lightweight porous residue after evaporation of that liquid."

This was witnessed and signed by W. B. Bennett and James II. Saunders, on August 21, 1952.

- Q. Who is Mr. Bennett? A. Mr. Bennett was one of my previous witnesses, who was a chemist working for Monsanto at that time.
- Q. And the other is Dr. Saunders? A. He was my group leader.
- (601) Q. You testified earlier that after this time when you had performed the first propylene oxide triisocyanate reactions, considerable additional work was done on this reaction.

Can you describe the avenues that this investigation took? A. Yes. By this time, people were getting more intrigued with isocyanate applications and we were getting more help, so, first I was working by myself. Then, I was assigned Marjorie Morris, who was strictly a technician, who did what I asked her to do but I did all the writing in the book.

Monsanto had what they called a co-op system, where chemists or engineers would go to school for a quarter and then work for a quarter—you are familiar with that, probably—and Billy Davis was such a co-op student and about this time he became available and he was assigned to our group.

So, I farmed this project out to him. I still supervised the work but he ran most of the experiments, starting about this time and was given his own notebook to fill out, which was our Exhibit AE.

- Q. What were the things that these people were doing with this reaction? What particular phase of it, for example, (602) did Billy Davis work on? A. He was working on almost specifically—maybe I shouldn't say almost, but he was working almost only on the solidification of the Aroclors by this reaction, mainly with triethanolamine.
- Q. Was there any foam work going on? A. Yes, there was foam work going on at the same time. There was adhesive work going on at the same time and there was what we call the isocyanate activity work going on, which

was our big problem, really, of being able to control these reactions, so we could get the same results each time.

This was still being done mainly by myself and Marjorie Morris.

Some time about this date, I don't know, we'll have to go back and look, I got another helper, Paula Combs, and she was given a lot of the activity work to do.

- Q. Will you please look at Mr. Davis' notebook, in particular pages 90101 through 90104. That is in Defendant's Exhibit AE. A. You see, these are dated January 6, 1953, the first date, so apparently that's when he became available.
- Q. I misspoke. That is your notebook, not Mr. Davis'. A. Which book is this, now?

Q. First give me— A. The first one is dated January 16, 1953, and we can take into account what I just said before about Billy's notebook because the system I was using with regard to Billy, since he was keeping his own notebook, I no longer had to write every little detail in mine, so in my notebooks on these pages I just put brief summaries of what he did in the week, in a week's work, and in my summary pages, as you can see, for example,

In other words, I was merely, for my own benefit, briefly summarizing what had happened on the work that

he was doing.

The same applies to 102, 103, 104 and perhaps some subsequent pages. These are summary pages of Billy's work.

Q. That work was gelling Aroclor:? A. Yes.

on 90101, I refer to his notebook 89901 to 89907.

(605) By Mr. Sweeney:

Q. Mr. Heiss, when we finished vesterday afternoon, we were at that part of your testimony where you were

explaining your summaries of Billy Davis in your notebook, which is Defendant's Exhibit AD.

Could you just briefly repeat what you have there so that the picture is complete for the testimony today? A. Billy Davis was a co-op and was eventually to be a technical person, so we encouraged him to write his own (606) notebook in contrast to Margy Morris, who was merely a technician. So, when he was assigned to this Aroclor project, he was given a notebook where he wrote in what he did at my direction.

Then in my notebook, just to make it convenient for myself, I made weekly summaries.

So, for example, in my notebook, 90101, is a summary of Billy Davis' notebook, 89901 to 89907, as indicated there about the middle of page 90101.

Similarly, 90102 is a summary of his notebook pages 89908 to 12, and so on.

Q. There are other similar summaries in that notebook, is that right? A. That is correct.

The Court: Excuse me.
Mr. Sweeney: Yes, your Honor.

The Court: Looking at 90101 and throughout, there are references to page numbers. On the top of 89101 is 82338-47. Now, I gather that refers to other references in your own notebook?

The Witness: Right.

The Court: If the numbers correspond with the Billy Davis notebook, they are references to his notebook, is that correct?

(607) The Witness: The part T wrote above that horizontal line was sort of tying together all previous work which T had done.

Now, the summary of Billy Davis' notebook starts below the line where it says, "Recent work is summarized below."

The Court: I understand that. What I am trying to make sure of is whatever numbers are referred to, if they are numbers that correspond with the Billy Davis notebook, like 89901, corresponding with his notebook, they are references in his notebook?

The Witness: That is correct.

The Court: The other numbers are references to your own notebook?

The Witness: Right.

Q. With the exception, Mr. Heiss, of the project number at the top of the page; what does that mean? A. We had for bookkeeping purposes various projects with different numbers, and 1080 was the number assigned to this project that we called isocyanate resins.

Q. Now, will you look at Defendant's— A. I might indicate one other thing, just so there is no confusion.

In my notebook summary pages, I sometimes make (608) comments and observations based on his work, but these observations will not be found in his book. I looked at his notebook pages, drew certain conclusions, and wrote those down in my notebook.

Q. Now will you look at Defendant's Exhibit AE, which commences with page 89901.

The Court: Is this the Billy Davis notebook? Mr. Sweeney: That is what I was going to establish, your Honor.

Q. Will you tell the Court what that notebook is? A. Well, this is the first page in Billy Davis' notebook and the first date therein is January 6, 1953, and he has a title up there on the first page, Preparation of Aroclor TDI Triols Adducts.

Then his first sentence is: "This is a continuation of previous work done on the solidification of Aroclor with TDI adducts." He starts his experiments.

- Q. What materials did you use so far as the condensates and the isocyanates are concerned? A. They are about the second or third sentence in the second paragraph. He says, "The triols to be used are ethylene and propylene oxide condensates of glycerine and triethanolamine," we can abbreviate that TEA, "prepared by Hector Flores."
- (609) Q. The isocyanate was? A. TDI.
- Q. Did Billy Davis work in the same laboratory with you? A. It was a laboratory that was sort of partitioned into two rooms, and the front was sort of an office, so he spent most of his time in the back part, which was the lab part, and I spent most of my time in the front part, which was like an office, except to go back to give him directions or observe what he did.
 - Q. You observed him doing his work? A. Oh, yes.
- Q. Did you observe him keeping his notebook? A. Yes. Yes, keeping a proper notebook is sort of a science in itself and we always try to train our people so they kept proper notebooks.
- Q. Now, you testified earlier that the ethylene oxide condensates that you had to have made were rather scarce. Why is it that you had Billy Davis work with these materials in the Aroclor project? A. Well, at the time that they became available, the PO adducts from Hector Flores, this is the project we were working on, so that is the one where they were first evaluated.

We did not want to use them up, for example, in (610) our project where we were working on isocyanate activity, as I explained yesterday: we preferred to do that work with commercially available materials, which we could replenish our supply any time we wanted.

Q. What were the results that Billy Davis got; what was your evaluation? A. Well, that is kind of hard to explain in a simple answer. He had a whole series of results and he made resins there, continuing the work that had previously been done in gelling this Aroclor up

into a series of products, some of which were hard and some were flexible, and some had bubbles in them which we were trying to get rid of.

Some were deliberately made into the foam form by using different catalysts.

Mr. Kelton: May I just interrupt you a minute. The Court: Yes, Mr. Kelton.

Mr. Kelton: Your Honor, I seriously question the relevance of this type of testimony by Mr. Heiss. Here he is dealing with January 6, 1953, and the prior art effective date of the Windemuth Patent is May 6, 1952.

Mr. Sweeney: For whatever it is worth, I was about to ask my last question on that subject.
The Court: All right.

Q. Were you satisfied with the materials that Billy (610a) Davis had made in this project? A. Yes.

Q. Was a patent application filed by you covering the work that you have described yesterday and today? A. Yes.

(611) Q. Mr. Heiss, T hand you Plaintiff's Exhibit 2, a certified copy of a file wrapper, and ask if you can identify it. A. I have to look at this carefully because there were several.

Yes, this is one of some applications that were filed about that time.

Q. That is a patent application of yours? A. Yes.

Q. I notice that there is a James H. Saunders named as a co-inventor in that application. A. Yes.

Q. Why was he a co-inventor? A. This application was the one which embodied several different approaches and in one of the subjects covered in this application he—I guess you could say he was a co-inventor in that he had suggested some of the catalysts which were used in a portion of the things included in this application.

- Q. Now, did that patent application ultimately result in a patent? A. Not in exactly the same form that it stands here, I don't believe. If I could see those patents again.
- Q. I hand you Plaintiff's Exhibit 1, Mr. Heiss, (612) which is your patent and the patent in suit, and ask you to look at the application number on the file history and see if they are the same. A. Where would the application number be on this? Oh, 373,036. Right. Yes.
- Q. That is the same as on the file history which is Plaintiff's Exhibit 2? A. Yes.
- Q. Dr. Saunders' name doesn't appear on the patent, Plaintiff's Exhibit 1. Why is that? A. Because this patent does not include everything which is in the original application and the part which was deleted, which is the part which was not present in this patent, is the part in which Dr. Saunders was—well, he was active.
- Q. What you mean is that the claims of the patent—A. The claims of this patent do not cover the invention in which Dr. Saunders was involved.
- Q. Who prepared the patent application, Plaintiff's Exhibit 2? A. The patent application?
- Q. Right. Who prepared your patent application? A. You are talking about this big thing now?
 - Q. Yes. (613) A. Mr. Rucker.
- Q. Where is Mr. Rucker now? A. Mr. Rucker is deceased.
- Q. He was the patent attorney at Monsanto? A. At the Anniston location, correct.
- Q. How did you get your information about the work you had done to Mr. Rucker? A. We generally wrote a memo which summarized the highlights and gave notebook numbers and sent this information to Mr. Rucker.
- Q. And in this case, did you prepare such a report? A. Yes.
- Q. Now, a moment ago you mentioned a number of patents had issued from that application. A. Yes.

Q. Do you know how that happened? A. Not exactly. It was something done for legal purposes which I never exactly comprehended, but all I know is that this application was split up into different patents and that's about the most light I can shed on it.

Mr. Sweeney: Mr. Clerk, will you mark this as Defendant's Exhibit AI?

(Defendant's Exhibit AI marked for identification.)

- Q. Mr. Heiss, I hand you a copy of a patent, (614) No. 3.143,517, which has been marked as Defendant's Exhibit AI, and ask if you can identify that as one of your patents? A. Yes.
- Q. I call your attention to the first paragraph of the patent, Defendant's Exhibit AI, where it says that:

"The application is a continuation of my application, serial number 373,036."

That is the application marked Plaintiff's Exhibit 2, am I correct? A. Yes.

Q. Would you please look at claim 2 of Defendant's Exhibit AI and just in general terms tell us what that claim deals with?

Mr. Kelton: If the Court please, again I question the relevance of this line of questioning.

Mr. Sweeney: I think I can explain it, your Honor.

The Court: Please do.

Mr. Sweeney: There has been testimony about the Aroclor work, there has been testimony about two-step reaction processes that Mr. Heiss ran, there has been testimony about the castor oil work that he did. All of these various projects resulted in patents.

I merely want to put in the record the fact (615) that these were other inventions, that they were

not just idle curiosity that Mr. Heiss was engaged in. Other patents issued on all of the work he did. These are all related and all come from the same application.

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That was the reason in my opening remarks I explained these various applications.

The Court: What relevance does that have to this controversy?

Mr. Sweeney: As the record stands now, I think there has been an attempt to show the Court that many things that are in the original application are not patented, are not covered by the claims of this patent.

I admit that they are not covered by the claims of the patent in suit, but they are covered by other patents, and that is all I was trying to do.

The Court: It would appear to me, particularly in view of the fact I am now submerged practically in exhibits, these are matters of record. I would think Mr. Kelton would be willing to concede that these are patents not in suit.

Mr. Kelton: I haven't had a chance to review this patent or the claims. I would be glad to concede that this number issued to Herbert L. Heiss and the date of issue, if that is what Mr. Sweeney wants to prove. If (616) he wants to have Heiss analyze the specification in the claims, I would question the relevance and materiality of it.

Mr. Sweeney: In order to solve the problem, your Honor, if I may simply read the numbers of the patent into the record, they are public documents, they are Mr. Heiss' patents, and if that's agreeable, that will suit me. The pents speak for themselves.

The Court: Is that agreeable?

Mr. Kelton: Yes. That is at least a short way. I still don't concede it. It has a virtue.

The Court: If the only purpose of this is to show that these other patents exist, that would be a short way to get it in the record.

Mr. Sweeney: With the understanding they would not be exhibits but that if I wish to refer to them in a post-trial brief I can do so.

The Court: All right.

Mr. Sweeney: The first is the patent I have already marked, 3,143,517, issued to Herbert L. Heiss August 4, 1964; the second is U. S. Patent 3,226,345, issued to James H. Saunders and Herbert L. Heiss December 28, 1965; the next is U. S. Patent 3,268,488, issued to Herbert L. Heiss August 23, 1966.

(617) Q. As a result of the work you did in the 1950s, Mr. Heiss, what did you learn about the differences between linear ethylene glycol and propylene glycol urethane and the branch ethylene oxide and propylene oxide condensate urethane? A. Well, in general, the branch materials gave superior polymers especially in the foam area to the linear ones. That was one general point.

And the other was that in comparing the propylene oxide to the ethylene oxide types, the ethylene oxide types were so water sensitive, as I mentioned yesterday, that the foam would change in firmness according to the humidity, according to how much moisture they picked up, whereas the propylene oxide foams were much more resistant to moisture and remained more constant under varying conditions. They were more practical materials.

Q. How great a difference was there between ethylene oxide foam and propylene oxide foam? A. I was surprised at the extent of the difference. One would expect that the polypropylene glycol would be a little less sensitive to moisture than the ethylene, but the difference was black and white, actually, that the polyethylene glycol materials were not practical materials for ordinary use, as,

per se, in the foam cushion or something (618) of that sort, whereas the polypropylenes were eminently successful.

- Q. Are you still doing research in the urethane field, Mr. Heiss? A. Yes.
- Q. Do you keep up with urethane developments generally? A. Yes.
- Q. The work that you did back in the fifties generally involved flexible urethane? A. Yes, mostly.
- Q. Do you know how flexible urethane foam is made commercially today? A. Yes.
- Q. How is it made? A. I presume you are interested only in the type that is made from polyethers, which is the bulk of the market. For example, the types nowadays used in cushioning and mattresses and automotive uses are based on polyether foam and as far as I know, at least, 80 and probably some higher percentage of all that flexible foam is made from a reaction of TEI and polypropylene oxide—let me say this correctly—propylene oxide condensates of triols such as glycerine or trimethylolpropane.

(619) Q. Did you ever make a presentation about your work to any commercial organization? A. Yes.

Q. When was that? A. I don't remember the exact date but it was at the ACS meeting I think in 1953.

The Court: ACS-

The Witness: American Chemical Society.

The Court: I thought so.

- Q. Do you remember the month? A. It was in the fall. Let's see, they are usually in September, right after Labor Day.
- Q. Do you know Dr. Windemuth? A. I have met him recently.
- Q. When did you meet him? A. After Mobay was formed I was sent on a trip to Germany to become familiar with some of their information on adhesives and

Herbert L. Heiss, for Defendant, Cross

coatings, and that is when I met Dr. Windemuth, since he was one of their main chemists working on adhesives and coatings at that time.

- Q. Do you remember the year you made that trip? A. That was either 1954 or 1955.
- Q. Did you know anything about Dr. Windemuth in 1951? (620) A. No.
- Q. When did you learn that he had done some urethane work? A. Well, when Bayer was formed he was presented as one of the persons I should talk to about ester-based polyethers—ester-based adhesives and coatings.
- Q. And about what time was that, the year? A. That was the visit I made over there in 1954 or 1955. 1955, I believe.
- Q. Do you know Dr. Charles Price? A. Only by reputation.
- Q. Why did you become aware of Dr. Price? A. He is quite well known in the industry, but specifically because we had that interference with him several years ago.
- Q. From your experience, what is the principal difference between ethylene oxide-based foam and propylene oxide-based foam? A. Repeat that, please.
- Q. From your experience, will you tell us what is the principal difference between ethylene oxide-based foam and propylene oxide-based foam? A. Well, again, as I mentioned before, this business of susceptibility to moisture. The ethylene oxide foams (621) are too susceptible to moisture and the propylene oxide foams are not.

(623) Cross Examination by Mr. Kosinski:

- Q. Mr. Heiss, are you a member of the American Chemical Society? A. Not at present.
 - Q. Were you a member in the past? A. Yes.
- Q. Would you please tell me the period during which you were a member? A. I don't know exactly. I started

in prior to 1951, I am pretty sure, and I dropped out about, I'd say, maybe 1960, 1962.

- Q. During the period that you were a member, did you receive publications which were published by the American Chemical Society? A. I received the C & E News edition.
- Q. Is the full name Chemical & Engineering News? A. Yes.

(628) By Mr. Kosinski (con't):

Q. Mr. Heiss, you testified yesterday concerning certain polyoxide adducts which you requested Hector Flores to make for you, is that correct? A. Yes.

Q. We had marked as exhibits Defendant's Exhibits AF and AG. Could you please tell me the date on which you received these materials from whomever you received them? A. No, I can't tell you that exactly except that it is between the date on the label there and the date they (629) were first used in our notebook.

Q. The label on each of these exhibits hears the date August 5, 1952. You testified that—rather, Mr. Flores testified he made two other samples for you, namely, the samples which he prepared, according to his notebook, by a particular reaction of glycerine with 3.1 and 13.2 mols of propylene oxide.

Do you recall the date on which you received those materials? A. Not exactly, but my recollection was that he made these four materials for us and didn't give us any of them until they were all ready, and he gave me the dates yesterday on which they were prepared.

(632) By Mr. Kosinski (con't):

Q. There has been testimony that you requested Mr. Flores to make these materials some time during the

latter part of July of 1952, is that correct? (633) A. Was it July? Whenever the record indicates.

Q. When you yourself actually went up and asked Flores? A. No, I did not ask Flores. I asked the group leader, Satkowski. I said, "We would like to have these materials. Can you make them for us?"

He said, "We are in the detergent business," and they had other things they were supposed to do. He said, "These are propylene rather than ethylene oxide. We will try to work them in when we can."

So it was left at that. We turned our interest to other things while we were waiting.

As Flores mentioned yesterday, I waited and waited and waited and nothing happened, and I went over to Satkowski again and said, "Where are our samples?"

At that point he jumped on Hector a little bit and they were made.

(636) By Mr. Kosinski (con't):

Q. Mr. Heiss, you have testified in considerable detail about a great number of experiments which you conducted in the years 1951, 1952 and 1953.

Now, isn't it a fact that the very first time that you ran an experiment with a condensate of propylene oxide and a triol or a tetrol was in September of 1953? A. Well, now you maybe can catch me on something. Is that something that was brought out yesterday in the testimony?

Q. I am not trying to catch you. It is the experiment which I referred to as the experiment conducted in September of 1953.

Mr. Sweeney: The witness say exptember of (637) 1952.

The Witness: What notebook page are you referring to?

Mr. Kosinski: 84531.

The Court: That is AC, isn't it?

Q. That would be notebook page 84531, which is in Defendant's Exhibit AC. A. Well, that is a page where we used it and unless it conflicts with something I said yesterday on an earlier page, yes, I guess that is the first time we used this material from Flores.

Q. Do you know of any other notebook page or other written record which reflects a run based upon a propylene oxide triol or a tetrol which is dated prior to September 19, 1952? A. I don't know of one, and since this was the one I presume we brought up yesterday, I hope there is not an earlier one that we are missing.

- Q. You make reference in the first paragraph of your entries on page 84531 to an adduct identified as Desmodur TH. Would you please identify that material for the record? A. Desmodur TH was a Bayer product which was sold—I believe that was a component to be used in the (638) coatings or adhesives. Do you want more identification?
- Q. Yes. Would you identify the nature of the chemicals which were utilized to prepare that product? A. I don't believe we knew exactly what it was, but we believed it to be a reaction product of three mols of TDI with one mol of trimethylolpropane essentially. There was some discussion as to whether or not there might not also have been a little bit of ethylene glycol mixed with the TMP before the reaction with the isocyanate. In other words, this was a way of making a moderate molecular weight triisocyanate adduct. This is the property which they still make, by the way.

Q. I would like to call your attention to a statement which is set forth under paragraph B which reads:

"More flexible than the TEA-TDI"-

A. Excuse me. Where are you now?

Q. Under paragraph B. It starts near the beginning of the fifth line in that paragraph. A. O.K.

Q. It reads: "More fiexible than the TEA-TDI-Aroclor resins."

And I would ask you if you made any other evaluation of the property which is described under paragraph B. A. Well, I couldn't say categorically no. There (639) may have been some evaluation on some subsequent pages.

Q. And did you record on page 84531 any other evaluations or comparisons with respect to the product or any of the products which are described on page 84531? A. Well, there are three products made: A, B and C. You are talking about B.

Q. I include all three in that last question, any other evaluations with respect to any of the products which are described on page 84531. A. Under A it says: "Changed to a clear transparent solid"—this is what I read yesterday—"containing many fissures. This looks like a good way to solidify Aroclor."

Then we went on and ran experiment B. This was the first time we used Aroclor. I shouldn't say "first." I don't know. This is where we used Aroclor instead of a benzene which we used in example A, and there we mentioned what you said plus "Will not melt or burn when removed from flame," which we thought was interesting. There is always an interest in non-flammable resins.

Now, we went down to example C, where we said: "Repeated B but cool during reaction to try to avoid bubble formation."

Q. What was the reason why you wanted or felt it (640) was necessary to avoid bubble formation? A. Well, if you are going to make a potting resin, which is one of the things we were trying to do, they are supposed to be homogenous and not contain air bubbles or gas bubbles.

Q. Did you record any comparison on page 84531 between any of the products there described and any product which was based upon an ethylene oxide condensate of a triol or a tetrol? A. Not on this page. Well, wait a minute. Not on this page.

Q. You make a reference to TEA-TDI-Aroclor resins. Would you please tell me the work to which that statement applies? A. Well, this applies to some of the previous work when we were solidifying Aroclors by reacting TDI with TDA. Without looking through a lot of notebooks, I can't tell you what numbers they were unless you have some clues, which I presume you have.

Q. I would refer you to page number— A. I can

refer you to a page I found now, 84526.

Q. The notebook entries to which I would like to now invite your attention is notebook page 84505 of Defendant's Exhibit AC, and that reproduces what appears to (641) be a note that you wrote to Dr. E. E. Hardy.

And before I ask you anything further about this page I would like to ask you to identify for the record who was Dr. E. E. Hardy? A. He was the assistant director at that time of the Phosphate Division Research Depart-

ment located in Anniston, Alabama.

(642) Q. Now, would you please describe for us the work just in general terms which led to your writing that letter to Dr. Hardy? A. In this note I refer to a report he had apparently written following a meeting to General Electric, indicating their interest in converting liquid dielectrics to solid dielectrics and I mentioned the fact that we had observed that some of our adhesive solutions in benzene had gelled and I make the comment there, without reading the whole page, in the fourth paragraph. I asked the question: "Would there he any advantage in attempting to cause a liquid Aroclor to solidify in this manner?"

Then more discussion after that. Do you want me to read the whole page?

Q. Yes, why don't you go ahead just so we have it in one place in the record? A. I will start at the top. "Solidification of liquid dielectric materials."

It is dated July 8, 1952.

"On June 13, 1952, I sent the following note to E. E. Hardy: I notice in your G.E. report that they are interested in converting liquid dielectrics to solids.

"You recall that by reacting disocyanates with polyols, etc., in such a ratio that the resulting product (643) contains OH as well as NCO groups, it is possible to cause 50 per cent benzene solutions to gel to rather firm masses.

"Would there be any advantage in attempting to cause a liquid Aroclor to solidify in this manner? Would this sort of mixture be able to compete with the polymerizable impregnants and offer a new field for liquid dielectrics."

That was the end of the quote.

Dr. Hardy indicated that such products would be useful if the gel was stable and had good dielectric properties.

Subsequent work (Notebook 82338) June 16 to 17, 1952, verified the above reasoning and reactions of equal molecular quantities of the dihydroxyoctachlorodiphenyl in Aroclor 1248 mediume resulted in the formation of a liquid which would gel to a hard glassy solid on standing overnight at room temperature. The liquid solid transition was reversible at about 70 to 80 degrees C. The solid has a dielectric constant of 5.3.

On the basis of the above work it can be seen that liquid dielectrics can be solidified with the use of isocyanate-polyol reaction products, thus making them useful as capacitor impregnants, potting resins and casting resins.

It is probable that a wide variety of dielectric (644) solids can be obtained by proper selection of isocyanates, hydroxyl compound, and liquid dielectric.

It is further possible that reaction of isocyanates with other active hydrogen compounds, such as amines, etc. yield similar results.

Then that was witnessed by W. B. Bennett and James

H. Saunders on July 8 and 9, 1952.

Q. Did you, commencing in the latter part of June 1952, embark on a research program having as its objective the solidification of liquid dielectric materials? A. Did I embark on a program in June?

Q. In the latter part of June, commencing the latter part of June 1952, having that as its objective? A. I don't know unless I look through the books. I know we were working on it about then. It didn't start out as that being the objective.

It started out when we sell the gellation of the benzene solutions. I don't know what—at exactly what point we started working for the potting compounds. It was apparently prior to this date of July 8, 1952.

Do you have any pages you would like me to look at?

Mr. Kosinski: Yes.

Your Honor, the reason for the delay is that I have (645) asked Mr. Sweeney if he would produce one of the original Heiss notebooks, which was not marked during their case as an exhibit. They are now looking for the notebook.

The Court: I will take the hint anyway from Mr. Kelton, and we will have a recess until 11:30. Mr. Kelton: Thank you, your Honor.

(Recess.)

Mr. Kosinski: Your Honor, I offer Plaintiff's Exhibit 55 in evidence.

Mr. Sweeney: I have no objection, your Honor. The Court: Received.

(Plaintiff's Exhibit 55 was received in evidence.)

Mr. Kosinski: I ask that there be marked as Plaintiff's Exhibit 56 the laboratory notebook of Mr. Heiss, hearing the page numbers 82301 through 82350, inclusive.

(Plaintiff's Exhibit 56 was marked for identification.)

By Mr. Kosinski:

Q. Mr. Heiss, I hand you your notebook, which has been marked Plaintiff's Exhibit 56, and ask you if this is another one of the notebooks which you kept while you worked at Monsanto? A. Yes.

(646) Q. Would you please identify the dates? A. The first entry is on May 5, 1952.

Q. The last entry? A. June 30, 1952.

Q. Was this the notebook in which you recorded your activities at Monsanto during the dates or in the period of May 5, 1952, through June 30, 1952? A. Yes.

Q. I direct your attention to the entries which appear on Page 82340 and I would ask you to describe the nature of the work, which is reported on that page? A. Well, this page refers back to 39, which refers back to a previous page, so these pages are all part of a series of reactions where we were looking at gelling agents and, in fact, this page is headed "Use of other adducts as gelling agents."

The date is June 18 and 19, 1952.

On this page various adducts are prepared as 50 per cent solutions in Aroclor 1242 and we react TDI with six different hydroxyl compounds.

Two were triols, glycerine and triethanolamine, and the others were diols.

- Q. The diols are identified by the symbols PPG, is that correct? (647) A. Yes.
- Q. Would you please consider \$2341, and indicate what the results were obtained from the experiments which are set forth on \$2340? A. The adducts were prepared, the solutions were prepared on \$2340, and then on \$2341 we looked at them after they had been stored overnight at either room temperature or in 100 degrees ('entigrade oven, and we make a series of observations.

In summary, without reading all the details, it says here, "B, Sample B, is by far the best solidified Aroclor prepared to date, especially in view of the fact that soluble adducts concentration is considerably below 50 per cent."

In other words, when we made A and B, some of the adducts separated out so the amount was still in solution at the time gellation occurred was less than 50 per cent.

- Q. Would you please identify for the record the character of the material that you referred to as triethanolamine? A. What do you mean "characterize"?
- Q. What is it? A. It is a liquid. It is a trihydroxy compound with a tertiary nitrogen in the center.
- Q. So T take it that of the various polyols screened, (648) you determined that triethanolamine was the best candidate? A. To date.
- Q. Did you do further work in connection with reaction of triethanolamine with TDI in Aroclors? A. Yes.
- Q. Would you please, if you would, just thumb through the balance of the pages in that notebook after Page 82341, and identify the pages on which you reported further work on your project of solidifying Aroclors with TDI, TEA adducts. A. 42—I will just read you the last two numbers—43, 44, 45, 46, because it is a continuation of 45, 47 is some analytical work, 48 and 49 are reviews of other materials so they are not pertinent here. 50—
- Q. So substantially most of your efforts in connection with your research activities during the period from June

16 rntil June 30, 1952, was spent on work on this project of solidifying liquid dielectrics with adducts of TDI and TEA? A. Yes.

- Q. Now, did your work continue on that project? A. I couldn't say for sure. We may have worked continually on that project, but that is not necessarily the only project we worked on.
- Q. Yes, I am speaking now of the project which had as its aim the solidification of liquid dielectrics with (649) polyol TDI adducts. A. What period of time are you talking about now?
- Q. Well, the last entry we have in your notebook, Plaintiff's Exhibit 56, was June 30.

Now, did you conduct further work on that same project and in that connection I would ask if you would turn to your notebook, which is marked Defendant's Exhibit AC. Which is the first page of that notebook which reports further activities on that project to which I referred in my previous question? A. Weil, the one you had me read before \$4505 doesn't report work, but it is the first page in that notebook pertaining to this subject.

Q. That is the page dated July 8, 1952? A. Yes.

- Q. What were you doing during the period June 3 to July 8, 1952? A. Well, one day I was probably celebrating the 4th of June, but we were now—well, on the pages 84501, 84502 and 84503, we are gathering together other materials for—let's see what this project is—more of a general—different approach, not working on this gellation at all, but in general trying to expand our knowledge of urethan compounds and urethane reactions. For example—
- (650) Q. I take it that according to the headings, which appear on Pages 84502 through 84504, you were concerning yourself with adducts of TDI with dihydroxy compounds? A. Aromatic dihydroxy compounds, right.

Q. Diols, those that contain two hydroxyl groups? A. Yes.

Q. Now, do you find any entries after July 8th in which you record further work which you conducted on this liquid dielectric solidification project?

The Court: You said after July 8th? Mr. Kosinski: After July 8, yes.

A. On 84501 there is a summary of the samples we had been looking at of the Aroclor TEA-TDI products, also carried over to Page 11, Page 12, there are some comments and apparently some experiments are run there.

Page 13 we start in earnest again in looking at—apparently here we are looking at different Aroclors.

- Q. Page 84510 and 84511 contain summaries of work that had been done previously on this project, is that correct? A. Yes.
- Q. What were you doing in the laboratory between July 8 and July 15, 1952, when you summarized the work previously done on this project? Just state the character of your work in general terms. You don't have to go into detail. (651) A. I don't know because we might have just been sort of reviewing things or maybe I was doing literature searches. There isn't must experimental work being done as indicated by this notebook.
- Q. Okay. Now, would you now indicate to me the first entry where you pick up the project and commence the conduct of further experiments. A. Would you repeat that or read it back?

Mr. Kosinski: I will rephrase it.

Q. Would you describe the first entry after July 8th which describes your further experimental work on what we have called the TDI-TEA project? A. That would be the one of July 15, that was a summary.

Page 12, we were—Page 13, 84513 is the one I mentioned previously where we were using the same TEATDI system, but now in this series of experiments we were using different Aroclors and some other materials,

halowax and santowax, to see what effect would happen or see what the effect was when the gellation occurred.

Q. I would ask you, in an effort to shorten this up, your Honor, if you would, please, Mr. Heiss, briefly review your notebook pages 84512 to 84526, which cover the period July 21 until August 20, 1952, and just state in general (652) terms the nature of the experiments which are reported on your notebook pages within that group? A. What page are you starting with?

Q. 84512 through 84526. A. I just told you what was on 12, 13 and 14—no, excuse me, I didn't. On 13, 13 we

have gone over.

14, another type of polyol we were looking at was castor oil and castor oil derivatives and we requested samples and these finally came in and on Page 514 we are starting to react TDI with different types of castor oil and castor oil derivatives.

Q. Was that in connection with the liquid solidifying project? A. No, these were a new class of materials we were looking at perhaps while we were waiting for Hector Flores' material.

515 is an idea sheet.

516 is entitled "Coating glass to make it shatterproof." We had a project going on that.

517 is a comparison of polyurethane films, which were prepared apparently on 84514, which were the castor oils.

84519 we are doing a little work with TDI, TEA Aroclor resin again.

(653) The same on 21—no, excuse me. 21 is back to castor oil again. Some revised reactions.

This is another thing that was consuming our time. When these experimental hydroxyl compounds came in, especially the castor oils, which were more or less functional fluids, there wasn't much known about their chemical structure and we, of course, were interested on what the hydroxyl content was of these materials as they were

received, so we were trying to evolve an analytical method so that we could take these commercial products as they came in, subject them to a chemical test to give us some idea on the equivalent weight, so we would know how much isocyanate to react them with.

Q. That was in connection with the castor oil material? A. Essentially, but we were trying to adapt it to any polyol material, but we started to work on the castor oils because we had no technical information on their hydroxyl content at all, on these impure types.

22 is a continuation of 20, whatever that was. That

was Aroclor solidification.

23—now we are looking at isocyanate adhesives again. You see we jumped around quite a bit.

24 is again this analytical problem, which we called isocyanate equivalent. We are still trying to determine a good analytical method.

(654) 25 continued from 23, so it is isocyanate adhesives.

26, we are back to the Aroclor solidification. It is an idea sheet. August 20. That is the last page you asked about.

Q. Right. Now, did you evaluate in connection with your dielectric solidification project any materials other than the triethanolamine TDI adducts during this period of July 20 through August 20, 1952? A. No, I don't helieve so. I think the only variable we had there was the different fluids, like that one page where we had the different Aroclors and those other two, the santowax and whatever else is on there. This work, I think, that we are talking about now, a lot of that was one to help us get an idea of exactly why these things were solidifying. We weren't exactly sure what was happening. We were pretty sure the Aroclor wasn't involved in the reaction, but it was still puzzling us why we would get a liquid adduct which then would polymerize into the solid material, what the mechanism was.

Q. So your comment as to flexibility, which you make under Paragraph B of 84531, was a comparison of that property in relation to your TEA-TDI Aroclor resins, which are reported in these notebook pages which we have been (655) speaking about, is that correct? A. No, it is not correct. Page 84531, we are trying to make an adduct of the type of Desmodur TH.

Q. I am addressing myself to the comment which appears in Paragraph B about which you testified earlier, namely, the statement, "More flexible than TEA-TDI Aro-

clor resins." A. Oh, yes.

Q. I understand from your testimony this morning that as a result of the experiments conducted, as reported on Page 84531, that your laboratory assistant, Billie Davis, conducted a series of experiments. A. I lose you there.

Q. I take it that from your testimony this morning that as a result of the experiments which you report on Page 84531 that a series of experiments were conducted by your laboratory assistant Billie Davis. A. I wouldn't put it that directly. As I recall his first entries were in January of '53, which was some three months later, so I couldn't say that they were conducted as a direct result of this page. By the way, I would like to point out something else here. You people are so concerned about how long it took us to work with Flores' material. You will notice there is no entries at all in this notebook from August 20 to September 61, so I might have gone on vacation then.

(656) Q. You mentioned the fact or you alluded to the fact that Billy Davis did not commence his work until January 6, 1953. Now, will you please tell us what, if anything, was done in terms of experiments from September, 1952 until January, 1953 which involved the reactions of polypropylene oxide triol adducts? A. You recall we had made some samples for sales on adhesives previously and I mentioned we had done some work on

coatings to make glass antishattering, and apparently during this period we were mostly working on that aspect.

We were beginning—this is the beginning, really, of that activity work, where we were concerned with why certain adhesive batches cured up faster than others.

Q. Will you please indicate the nature of the material which was utilized in formulating those adhesives during that period of time? A. I think we did most of this work with the polyethylene glycol 400 again of which we had a large supply.

Q. Those were the diols about which you testified earlier? A. Yes. I see on page 84536 I see Aroclor (657) mentioned. It refers to 84535. You see, we are reacting TDI with PEG 400 in Aroclor there. Apparently, these didn't gel up as did the previous materials, where we were using triethanolamine and other triols.

We state and make comments here that the viscosity of the sample increases even when they were heated at 200 degrees C., but they apparently did not solidify, as did the previous samples.

Any point to detailing that page for your benefic?.

Q. No, I just wanted you to make a general statement as to the nature of your activities and I think you have done that during the period I asked you about, namely, September 19, 1952 to January 6, 1953. A. I think these pages are a pretty good indication of the variety of all the work we were involved in at that time, that we didn't have any one project that we were working on.

Q. I would next direct your attention to Defendant's Exhibit AD. I understand from your earlier testimony that you summarized on pages 90901 and the following pages the experimental work that was conducted by Billy Davis with ethylene oxide and propylene oxide adducts of glycerine. Is that correct? (658) A. Well, at least it is with 90101. I see that comment made.

Q. I would just like to go through briefly, if I may, these summaries which you report in this notebook. You discussed 90101 and I would like to ask you about your entries on page 90191.

Did you make any entry on that page which reflected any evaluations which were performed or observed with respect to the hydrolytic stability properties of EO as compared with PO condensates of glycerine? A. No. I don't think we would in that instance where we are working with potting resins.

Q. I direct your attention to page 90102. A. Now, wait a minute. Hydrolytic to me means chemical decomposition by hydrolysis. You are referring, I suppose, to this business I was talking about of absorbing moisture.

Q. Let's call it water sensitivity. A. Okav.

No, we didn't, because, for example, if either one of these resins was dissolved in something like Aroclor 50 per cent, which is very hydrophobic, I doubt very much that in these types of materials that that would (659) be a big factor because the Aroclor is providing the non-sensitivity to the moisture.

- Q. In other words, they would both be equal in terms of that property? A. For practical purposes, I would say.
 - Q. I would like you to turn to page 90102.

Now, does that reflect further work done by Billy Davis? A. Yes.

- Q. On that project? A. Yes.
- Q. Now, do any of the entries on page 90102 describe any evaluation or comparison of the relative merits of EO versus FO glycerine condensates? A. For any reason?
- Q. For any reason. A. No. Obviously, no. I think these were all paid with PO. Let me read the page.

(Pause.)

A. There is nothing on this page that compares the two, but I don't know what observations were made in his notebook. Here, apparently, the PO ones were the best because it says here the glycerine condensate TDI adducts—this is in the second paragraph—and so on (660) and so on, so I presume now, without looking in his book, that he was using both the glycerine EO on the glycerine PO.

Let me look in his notebook. That is the best way to resolve this. You see, he is working on a few things, also.

For example, on his pages 89908 and 89909 they are not even concerned with this gellation of Aroclor. That was another project we had. We were trying to make a synthetic wax-like material, a substitute for carnauba wax.

On his page 89910 he apparently has a table summarizing a lot of reactions and the reactions involved glycerine plus EO and glycerine plus PO.

The left-hand column there indicates what the reactants were.

You notice on the following page, on page 911, he is now using only the glycerine PO series. He is running two series there from A to D using the high molecular weight material containing 13 PO per mole glycerine, and E through H, which are lower molecular weight of 3.1 PO per mole of glycerine

So without reading all this thing in detail, I would assume that the reason he only used PO on 11 was (661) they gave better products than the EO ones, which he used on 10, because he didn't bother to repeat those.

Q. Apart from your assumption, I take it that the properties of both the EO and the PO products, at least to the extent observed by Billy Davis, are as reported at the top of page 89910? A. Yes.

Q. I next ask you to look at page 90103.

Does that contain a summary of the work conducted by Billy Davis during the period January 19-23 as indicated? A. Yes.

Q. I take it that the work to which you referred on this page involved solidification of Aroclor fluids utilizing TDI and glycerine on 13.2 PO. I make that assumption on the basis of the first paragraph. A. What was your assumption that you made?

Q. I assume that they all involve glycerine PO con-

densates. A. Yes, that is what it says there.

Q. Would you please read into the record your conclusions with respect to that series of experiments? A. You mean you just want me to read the bottom, last

paragraph?

(662) Q. Yes, please. A. "Most of the resins prepared in this way, especially the more brittle ones, lacked toughness. The softer resins suffer from what might be likened to poor tear resistance. Possibly the use of MDI or other triols (castor oil, GER 108 resin) will improve the resin sufficiently."

Q. Would you please turn to page 20104.

That page, I take it, summarizes the work done by Billy Davis as reflected on pages 89924-29 of his note-book? A. Yes.

Q. Do pages 90104 through 90108 reflect your summary of further experiments conducted by Billy Davis on this Aroclor gellation project? A. Well, 90105 is an idea sheet, so that doesn't.

90106 is merely the notation that our lab moved again so that week was shot.

90107, part of that at least is on the gellation. I notice we are becoming again more concerned with the activity thing. I see some comments here to adjusting HCL content in the TDI. This was also concerned with this gellation thing. It was a factor in that also.

(663) So without my looking at his notebook pages, I can't tell exactly what all the details were, but I see here in the last paragraph on 107 the comment made that casting of resins for electrical testing, and so forth and so on.

Q. I invite your attention to page 90110 and ask you if you would read into the record the statement which you made under the heading "Conclusions." A. "In view of the difficulties encountered in the preparation of bubble-free resins, this project will be discontinued in favor of others that promise to be of shorter duration. It is felt that this solidification of fluids is of potential value, however, and the work will be resumed when we know more about isocyanate reactions."

Q. Did you, Mr. Heiss, in the period from March 9, 1953 to August 7, 1953, ever conduct an experiment in which you reacted a condensate of propylene oxide with a polyhydric alcohol containing 3 or 4 OH groups with a polyisocyanate? A. Well, for me to answer that definitely I would have to look through all those pages, unless there is a page somebody can suggest that I should look at.

Q. I can tell you that I have looked through them and I haven't found any such experiment, but I (664) would like to have you tell me if you know of any record in your notebooks which would indicate that you ever did in fact conduct such an experiment.

The Witness: Am I allowed to discuss things with you? I would like to ask a question.

A. Well, of course, when we were going through this we have gone through all these notebooks and we know what pages are pertinent. If there is some way Mr. Sweeney can tell me what the page is it will save me looking through all these pages.

(667) Mr. Kosinski: I ask that there be marked as Plaintiff's Exhibit 57 a laboratory notebook bearing the numbers 88451-88500.

(Plaintiff's Exhibit 57 was marked for identification.)

- Q. I hand you Plaintiff's Exhibit 57 for identification and ask you if this is another one of your laboratory notebooks? A. Yes.
- Q. Will you please identify the dates during which you made entries in that notebook? A. The first page dated November 18, 1952. The last page dated April 8, 1953. We need another notebook.
- Q. Would you please consider the entries between the dates March 9, 1953 and the last entry and tell me if any of those entries reflect any experiment you performed which utilized a condensate of propylene oxide and a polyhydric alcohol containing 3 or 4 hydroxyl groups? (668) A. The latter part of this notebook, all the experiments are entitled "Isocyanate activity," which is that problem we had of controlling the reaction, so I doubt very much that any of our special triols were used in that work.

There may be another notebook covering a similar time period because about this time we had so many projects going I sometimes kept two or three notebooks, one project in each one.

Q. But that notebook does not reflect any experiments based upon the use of a propylene oxide adduct? A. No.

Mr. Kosinski: I next ask that there be marked as Plaintiff's Exhibit 58 a notebook identified by the numbers 92401-92450.

(Plaintiff's Exhibit 58 marked for identification.)

Q. Mr. Heiss, I hand you a notebook marked Plaintiff's Exhibit 58 for identification and ask you if this is another one of your laboratory notebooks? A. Yes.

Q. Would you please consider the entries in that note-book from March 25, 1953 to August 7, 1953 and (669) state whether or not any of those entries reflect any work that you did based upon the use of a condensate of propylene oxide with a polyhydric alcohol having 3 or 4 hydroxyl groups? A. I probably can save time here and say probably not because I notice this is a notebook dealing with isocyanate customer problems, where we were working with a response back from some of the samples we had sent out, and that did not apply to the Aroclor gellations.

I will look to make sure, but I would be surprised

if there is any of that in this book.

Q. I am not restricting my inquiry to Aroclor gellations. I am speaking about any experiment for any purpose based upon the use of a propylene oxide type of triol. A. Oh, no.

Mr. Kosinski: I ask that there be marked as Plaintiff's Exhibit 59 a laboratory notebook bearing the numbers 92651-92770.

(Plaintiff's Exhibit 59 was marked for identification.)

Q. Mr. Heiss, I hand you the notebook which has been marked Plaintiff's Exhibit 59 for identification and (670) ask you if this is another one of your laboratory notebooks. A. Yes.

Q. Would you please tell me the period covered by this notebook. A. The first date is April 8, 353. The last date is August 14, 1953.

Q. Now, would you please consider the various entries in that notebook and tell me if any of those entries relate to a polyurethane product which is based upon the use of a condensate of propylene oxide with a polyhydric alcohol containing 3 or 4 hydroxyl groups? A. Well, up through page 83 all the pages are headed "Isocyanate activity," so I doubt if we used our special product there.

We were working with the linear materials. We weren't trying to make polymers so much as understand the reaction.

Q. Linear materials—are you referring to the fact that the polyol intermediate there utilized contained two hydroxyl groups? A. Yes, most of this work seemed to be done with our old workhorse PEG 400.

In fact, I notice there is apparently a (671) misleading there. Page 84 is entitled "Tsocyanate application," but it is the same job number. I would presume this whole book is concerned with isocyanate activity. All the remaining pages are headed "Tsocyanate activity."

This was a problem we felt we had to solve before we could really make reproducible polymers of any type in the sense of really getting a nicely controlled reaction.

Q. I take it you don't find reported in that notebook any experiment based upon the use of a condensate of propylene oxide with a polyhydric alcohol containing 3 or 4 hydroxyl groups? A. No.

Q. You referred this morning in your direct testimony to the fact that the way the patent application work was handled when you worked at Monsanto is that you would communicate information concerning your work to a Mr. Rucker. A. Yes.

Q. What form did those communications take? A. Well, many forms. We were, of course, putting out periodic progress reports which varied from time to time. Sometimes they were monthly, sometimes they were bimonthly and sometimes they were quarterly. The (672) headquarters in St. Louis would keep changing around what plan they wanted to use.

Anyway, these were periodic reports indicating how the work was coming along. Then if the work looked very interesting and seemed important that we should get it out into a sales brochure or something to help get other people interested in isocyanates we might make what

was called an interim report indicating the work could be summarized, but we didn't consider it completed. These could be used by the sales department to put out a little preliminary market bulletin.

Another form of report that we had was a shortform report or a long-form report, which would, when the job was done, be that you wrote it up that way.

So these were put out. But then generally if we had an idea that we thought was usually good or should be considered for patentability, we would write either an idea sheet or a memo to Mr. Rucker indicating and emphasizing: Here, please consider this.

He would have access to all the other information which had been published on that project in addition to this memo.

Q. Did you provide Mr. Rucker with a memorandum setting forth the information which was developed on the Aroclor solidification project? (673) A. I don't recall that I gave him a memo dealing only with that, but I recall writing memos to him indicating our success with these urethane products of all types and left it to him to decide how he was going to handle it.

Mr. Kosinski: T ask that there be marked as Plaintiff's Exhibit 60 a memorandum entitled "Solidification of fluids with isocyanates."

(Plaintiff's Exhibit 60 was marked for identification.)

Mr. Kosinski: This memorandum, I might note for the record, is dated February 13, 1953.

By Mr. Kosinski:

Q. Mr. Heiss, I hand you the memorandum which has been marked as Plaintiff's Exhibit 60 for identification and ask you if that is the memorandum to which you referred. A. This is one of some. I recall sending an-

other memo in some more general form, but, yes, this is one of them.

- Q. Did that memorandum set forth what you believed at the time you prepared it to be your novel ideas? A. I would say we should not call it necessarily what I consider novel ideas. It merely presents our (674) observations. I left it for him to decide whether they were novel or not.
- Q. The reason I used the word "novel" is because of a statement made in the last paragraph on page 1 which reads:

Although the reactions and processes will undergo considerable refinement, we would like to have some coverage on the original novel idea of solidification of fluids by polymerization of materials containing unreacted NCO groups dissolved therein."

Now, I would like to ask you a couple of questions about this memorandum.

I will first ask you if this memorandum mentions anything about any comparison between the properties of polyurethane products formed by reacting PO versus EO condensates of polyhydric alcohols containing 3 or 4 hydroxyl groups with polyisocyanates. A. Well, this memo apparently does not. It refers to a whole succession of notebook pages, which I presume are included in the ones you have already gone over, so, no, this memo in itself does not mention that comparison.

Q. Is there any statement made in the body of the memorandum with respect to the propylene oxide or ethylene oxide condensates? (675) A. Well, I don't know exactly. It says in this first paragraph, about the second or third sentence:

"Depending upon the type of product desired, the diisocyanates and polyols (or other active hydrogen compounds) are reacted as described in H. L. Heiss patent application, serial No. 300,968."

So what is contained therein I do not recall.

Q. I also note on page 2 there is a heading—it is en-

titled "Original idea and extensions thereof."

I take it that the notebook pages which you listed there at that time contained the information which you believed then to communicate the "Original idea"? A. I presume that these pages contain or these numbers are the pages which we had witnessed and I would consider to be idea sheets, which were possibly the original idea, plus whatever we added on as we saw additional results.

(685) Mr. Kosinski: I offer Plaintiff's Exhibit 61 in evidence.

Mr. Sweeney: Your Honor, in this case I think I am entitled to ask the purpose of the exhibit before it goes in.

Mr. Kosinski: Well, I think the purose is perfectly plain. This is a correlation—

The Court: Not to Mr. Sweeney.

Mr. Kosinski: This is a correlation of the examples by which the invention is allegedly exemplified in the patent and suit and this correlation identifies where in his notebook records and in what connection and for what purpose and with what results the experiments, under line experiments, were conducted.

The Court: Mr. Sweeney?

Mr. Sweeney: Well, am I to understand that there is some dispute as to whether or not the examples were performed as recited in the notebook.

Mr. Kosinski: No. Mr. Sweeney, there is no dispute.

The Court: I don't believe there is a dispute. Isn't what you are looking for in terms of these

examples whether or not in the notebook any of the examples evidenced the experimentation?

(686) Mr. Kosinski: Well, that is not the point. The point we are trying to make for the purpose which I am going through this testimony is that in the underlying experimental program in connection with which these examples were generated, your Honor, the sole purpose and the sole objective was to solidify plasticizers.

In other words, the plasticizing component was there in the most experiments in very large quantities and this was the underlying concept which led to all of this work, namely, to solidify the plasticizers and, therefore, I think that—

The Court: I want to ask you what difference does it make if that was the purpose of it if, in fact, the experiments were entered in an advancement of the art pursuant to what's been related here?

Mr. Kosinski: Well, the point is that his sole aim at that point—

The Court: You might accidentally come across something.

Mr. Kosinski: It doesn't show any appreciation in the record, if he did.

The Court: What I am saying is that the fact that he was involved in plasticizing or, rather, if that (687) was the purpose of it, if he did in fact experiment with the substances which I gather that there is some contention as to whether or not he did, or made any advancement in the art during that time, the fact that he was looking for one thing and found another, that wouldn't be any delimination of the advances that he made, would it?

Mr. Kosinski: Well, I think one aspect of this is that in the terms of what he was looking for,

in terms of what his work was devoted to, he himself had believed that the only thing he made that was of any significance was something which was based upon a three-element system and that's the purpose for which we have gone through this correlation, at least one reason we feel this is relevant.

(689) By Mr. Kosinski (con't):

Q. Mr. Heiss, I hand you a copy of your patent, 3102875, Plaintiff's Exhibit 1, and ask you if there are any statements contained in the specification with respect to any relative comparison between polyurethane products made using condensates of PO and a polyhydric alcohol containing three or four OH groups on the one hand and those same condensates made using ethylene oxide.

The Court: What's the question? Was there any comparison?

Mr. Kosinski: Any comparison between the relative properties of EO and PO.

A. Now, you realize that when you ask it that way, since I don't remember all these patents, you are asking me to read this, right?

Q. Well, read the specification. A. Which is what, the first part up to the examples?

Q. No. Up to—well, let's take it in two steps, yes. A. Good.

Mr. Sweeney: Your Honor, the patent speaks for itself. If it says it, it says it; if it doesn't, it doesn't.

The Court: Why do we need that?

(690) Mr. Kosinski: Well, your Honor, Mr. Sweeney seems to emphasize throughout these proceedings that at least one major basis for the invention is

the fact that there are a great many attributes which attach to a propylene oxide adduct as compared to an ethylene oxide adduct.

Now, I think it's fair to ask this witness if what Sweeney emphasizes as the basis for patentability

is disclosed in the patent in the issue.

The Court: But if the witness is able to answer the question, and the witness says he has a lot of patents to read through, it seems to me that if it is disclosed, it will be disclosed here.

Your theory is that it is not?

Mr. Kosinski: I am only asking him as to this exhibit, just as to this exhibit.

Q. Are you familiar with this patent, Mr. Heiss? A. Yes, sir. I know there is such a patent and I know I am the inventor. I don't say I sit around reading it every day, no.

I don't know what's written on there, every word.

Q. Well, do you know whether or not there is any statement contained in the specification with respect to any advantage attaching to the use of an adduct which is a condensate of propylene oxide with a triol or a tetrol? (691) A. I know that we knew there were advantages. Now, if you want me to say whether it's stated in here, I repeat, I'll have to read it, because I am not going to say it's not in here just on the basis of my memory and then, perhaps, it is.

The Court: It seems to me that, under the circumstances, unless you really want his statement on it, I don't see what importance that would be one way or the other, at this point, because the patent speaks for itself.

Q. Do you know of any advantage now? A. Certainly That's what we have been talking about for how long? I have lost track of time.

We keep repeating the difference between propylene oxide and the ones made from ethylene. We can demonstrate it to you if you wish.

Q. But you are unable to say on the basis of your present recollection whether or not any of the advantages are specifically set forth? A. That's right. One reason being that this is a fragment of an original application and I wasn't that concerned how the attorneys split this up. That was their business, not mine.

Q. I would like to have you, if you would, please, look at claim 3, and first read it to yourself. That's (692)

Plaintiff's Exhibit 1. A. Yes?

Q. Now, you will note that the material set forth as ingredient 1 is defined as a condensate of propylene oxide and a polyhydric alcohol having three to four hydroxyl groups.

Now, I would ask you if you would please point out, if you can, where in the specification is found a description, a chemical description, of a condensator propylene oxide with a polyhydric alcohol having four hydroxyl groups. A. This is the same thing in which we engaged in a correspondence and we sent you a letter pointing this all out and I'll have to find it again.

Column 2, starting about line 25 or 26—tetrahydroxy compounds, such as pentaerythritol, et cetera, alkylene oxide condensates or pentaerythritol, et cetera, and so

on and so on.

Q. Mr. Heiss, I put before you Plaintiff's Exhibit No. 2, which is the file wrapper of your Patent Number 3102875, and direct your attention to pages 130-131, which is identified as a "oath," and ask you if that is your signature which appears on page 131? A. Yes.

(693) Q. Did you believe that all of the statements contained in this oath were true and correct at the

time that you signed it? A. Yes.

Q. Mr. Heiss, I place before you Plaintiff's Exhibit 27 and direct your attention to the preliminary statement

which is the first document contained within this exhibit and I ask you if that is your signature which appears on the third page, numbered 8, of this exhibit. A. Yes.

Q. Did you believe that the statements set forth therein were true and correct at the time you signed them? A. Yes, sir.

Mr. Kosinski: I ask that there be marked as Plaintiff's Exhibit 62 a document entitled "Short Form Report," which bears the date at the lower left-hand corner May 19, 1954.

(Plaintiff's Exhibit 62 marked for identification.)

Q. Mr. Heiss, do you recognize Dr. Saunders' signature at the bottom of the first page of Plaintiff's Exhibit 62? A. Yes. It looks like his signature.

Q. Were the experiments which are described and (694) summarized in this report experiments which were performed by you or by somebody acting under your direction and supervision? A. Yes.

Mr. Sweeney: Your Honor, may I say that this report is dated May 19, 1954, which was long after the application was filed and hardly relevant to the application and I did not bring this one up.

Mr. Kosinski: Well, it does cover a period which includes the period from April 10, 1953. You see, at the top it says "Work started April 10, 1953," so it does include work during a period prior to the filing date and, I think more importantly, it reflects experimental activities by Mr. Heiss and his co-workers as a result of which they determined that both ethylene oxide and propylene oxide derived foams were greatly weakened by water.

(695) The Court: Well, I am not going to restrict the questions. If there was an objection, it's overruled.

Herbert L. Heiss, for Defendant, Re-direct

Q. Mr. Heiss, I direct your attention to the summary and I would ask if you would read into the record the first paragraph under the heading "Summary." A. "Both rigid and flexible foams were prepared from diisocyanates and a variety of polyols. Those foams prepared from ethylene oxide and propylene oxide derived polyols were greatly weakened by water. Foams from hydrogenated castor oil and polybutylene glycols were not affected greatly by water. All of these foams were weaker than the best diisocyanate polyester foams of comparable density."

(697) Re-direct Examination by Mr. Sweeney:

Q. Mr. Heiss, do you still have Plaintiff's Exhibit 62? I wonder if you would look at it and tell me what kind of foams are reported on in that report. A. Both rigid and flexible foams under the conclusions on page 1.

Q. I am more interested in what kind of dissocyanate and polyols were used. I think the body of the report tells you that. A. Well, we used both TDI and MDI and we used glycerine PO adducts in conjunction with linear diols. (698) We used castor oil, which is a triol and we used a modified form of castor oil on notebook No. 99976-2.

We used some linear polypropylene gycols only.

From this table 2 on page 6, if that's a representative summary, there are no examples where the branched glycerine PO adduct was used in a foam by itself. It was only used in conjunction with the linear material.

I don't know what the situation is with regard to the specific examples in the notebook, however, but looking at 2 no foams were reported there that were prepared only from a glycerine adduct of propylene oxide and an isocyanate.

Clelle W. Upchurch, for Defendant, Direct

Q. Will you look at notebook page 90103, which would be in Exhibit AD.

In the work that you were doing there, if I understand your report, you were not using Aroclor; is that correct? A. Well, if the similar page is correct, it's correct. It says in the first sentence, "Flinds other than Aroclor were solidified," et cetera, et cetera, right.

Mr. Sweeney: I have no further questions, your Honor.

(699) The Court: Mr. Kosinski, anything further?

Re-cross Examination by Mr. Kosinski:

- Q. On that notebook, page 90103, as I understand the summary, in the experiments which are summarized, other plasticizer-type materials were used in place of the Aroclor; is that correct? A. Yes.
- Q. And they, like the Aroclors, were there for the plasticizer function? A. Well, you can call it that. They were the fluids which were solidified.

(703) CLELLE W. UPCHURCH, called as a witness, being first duly sworn, testified as follows:

Direct Examination by Mr. Sweeney:

Q. Mr. Upchurch, where do you reside? A. Arlington, Virginia.

Q. What occupation are you engaged in in that area? A. I am a patent attorney. I have an office in Arlington, Virginia.

Q. Would you state your professional background, please? A. Just professional or with education?

Clelle W. Upchurch, for Defendant, Direct

Q. Well, you can interlace them, if you would. A. I graduated from the University of Missouri in 1938 with a major in chemistry.

(704) I went to work for Olin, Mathieson Chemical Corporation, was a chemist for eight or nine years and

then moved into the patent department.

I went to law school at St. Louis University and became a member of the bar of the State of Missouri in 1956; later became a member of the bar of the District of Columbia and, still later, became a member of the bar of Virginia.

Q. Are you admitted to practice before the United States Patent Office? A. Yes.

Mr. Sweeney: Your Honor, this is a matter I might explain. In order to practice in the Patent Office, you must take another test, like another Bar Exam. It's the only federal agency that has that.

The Court: I begin to understand why.

Q. Mr. Upchurch, if I may, I'll hand you Plaintiff's Exhibit 1 and ask you if you can identify that as the Heiss patent which you had something to do with. A. Yes.

Q. I'll hand you Plaintiff's Exhibit 2 and ask you if you can identify that as a certified copy of the file history of

the Heiss patent. A. Yes.

(705) Q. If you would, please, look at page 43 of Plaintiff's Exhibit 2. A. Let's be sure of the number. There are three numbers on the bottom of the page.

Q. You need to go by the written-in numbers.

Would you tell us what that page is, page 42? A. Well, it's an associate power of attorney from Don Haefle, who was one of the attorneys of record at the time.

Q. Who does Mr. Haefle give the power of attorney

to? A. He gave it to me.

Q. What's the date of that document? A. Well, it was received in the Patent Office on March 8, 1957.

('lelle W. Upchurch, for Defendant, Direct

Q. Is that the beginning of your active role in the prosecution of the Heiss application? A. Yes.

Q. If you will, look at the amendment that begins at page 60. It goes through page 79.

Is that an amendment that you prepared? A. Yes.

Q. Would you explain to the Court just what an amendment of this type is? I mean, what it formally is (706) doing insofar as the prosecution of this application is concerned. A. Well, we had received an office action dated July 31, 1957. Apparently, the Patent Office has pointed out reasons why the claims in the application were not considered patentable.

There were rejections based on prior art. Then, the purpose of an amendment is to—we call it an amendment and it is a response to the office action and it is truly an amendment, if you change the claims or, if in some way you add new claims, but it is a response to the office action.

By statute at that time it had to be—you had to respond within six months and it's your response to his rejection, trying to persuade him that you do have patentable subject matter.

The Court: The office action means that the Patent Office has taken some action in regard to this and you have to respond to it?

The Witness: Right. An examiner had studied our claims and decided that at that point there were claims that were not allowable.

Q. Mr. Upchurch, would you look at Claims 35 and 53, which you added by this amendment. (707) Would you read them? I think it would expedite matters. A. All right.

(Pause.)

Q. Would you explain to the Court what those two claims cover? A. Well, Claim 35 is directed to the re-

Clelle W. Upchurch, for Defendant, Direct

action product you obtain when you react a triol, which would be glycerine or some other trihydric alcohol, and in this case it's the alkylene oxide condensate of the organic trihydroxy compound.

In other words, it would be glycerine, in which you have condensed propylene oxide to make the long chain

branched molecule you have been talking about.

It's reacting that with isocyanate. The isocyanate here is identified as R(NCX)n. X could be either oxygen or sulfur, so it could be either an isocyanate or a triisocyanate. n is at least two; that means it's at least a diisocyanate.

It could be a diisocyanate or a triisocyanate or some other isocyanate having more than one NCO.

Q. Is that a customary way to write chemical formulas in patents? A. Yes.

(708) Q. Is there any mention of a solvent in that claim? A. No.

- Q. Would you mind, Mr. Upchurch, doing the same thing with Claim 53? A. Well, Claim 53 is directed to the method of making a product of the type we were claiming in Claim 35. You are reacting the condensation product of an alkylene oxide and an organic trihydroxy compound with a diisocyanate or triisocyanate or the thiosocyanate.
- Q. When did you file that amendment, Mr. Upchurch? A. It was received in the Patent Office on January 27, 1958.
- Q. I think I forgot to ask you if there is any mention of solvent in Claim 53. A. No, there is not.

Q. Is propylene oxide an alkylene oxide? A. Right.

- Q. At the time you filed this amendment, did you know about the Price patent? A. No.
- Q. Do you know when the Price patent issued? A. I am not sure of the date. I did know it at the time it issued.

('lelle W. Upchurch, for Defendant, Direct

The first time I saw the price patent or knew (709) about the Price patent was when I saw the claim published in the Patent Office Gazette.

Q. The record will show that the patent issued December 30, 1958. A. Well, then, I knew about it some time after December 30, 1958.

Q. How long did Claims 35 and 53 stay in the Heiss application? I think if you look at page 152, you will be aided. A. Well, Claims 35 and 53 were among those cancelled in the amendment filed on September 26. It looks like 1962.

Q. Which was after the termination of the Heiss-Price Interference? A. It's not stated just that way here in the file, but I notice that on June 19, 1962, we asked for an extension of time to file our brief on appeal and we wouldn't have been filing a brief on appeal during the Interference.

(710) By Mr. Sweeney:

Mr. Sweeney: That is correct.

Q. Now, can you tell us how the interference between the Price patent and the Heiss application came about and I mean in general terms, I don't want you to go into great detail? A. Well, after I saw the claim in the Gazette and got a copy of the patent, we copied the claims of the Price patent and requested an interference.

Q. Which was instituted? A. Yes, it was.

Mr. Sweeney: Your Honor, this is another term of patent art. When a patent has issued that you think you can get in interference with, then you actually copy the claims out of the issued patent, put them in your application by way of one of these amendments and then you say to the Examiner, "now, there are claims to the invention in both places, declares the interference."

This is what happened here.

Q. Mr. Upchurch, I hand you Defendant's Exhibit I and ask if you would explain to the Court what that is in general terms only? A. Well, in general it is a motion filed by Mr. Price after the interference had been declared and in the (711) motion he is asking the Patent-Office to dissolve the interference and in effect declare that there is no interference between our application and—or the patent of Price.

Q Can you explain to the Court the two grounds on which Dr. Price relied in making this motion? A. Well, Ground A, the Party Heiss has no right to make the

claims forming the counts of the interference.

As I recall that was based only on the contention that the patent claim had a molecular weight limitation of at least 600 and Price contended that there was no basis in the Heiss specification which would support such a molecular weight limitation.

Q. What was Ground B?

The Court: The molecular weight limitation was in the Price patent?

The Witness: Right. It is the molecular weight of the polyol.

The Court: All right.

Q. I think if you look at Page 11 of the motion you will find it. A. Ground B was the contention of Price that Heiss was not entitled to claim an invention except one in which a plasticizer was included and one of the components of the product or of the reaction mixture. (712) Q. Would that he the Aroclor material? A. Right,

Mr. Sweeney: Mr. Clerk, may I have Defendant's Exhibit J?

Thank you.

Q. Mr. Upchurch, I hand you Defendant's Exhibit J and ask you to briefly explain what that is. A. This is a decision on the motion to dissolve.

Q. This is the Patent Office decision? A. The Patent Office decision by the primary examiner on the motion that had been filed to dissolve the interference.

Q. What was the decision? All I am saying is did

he deny it? A. He denied it.

Q. What did he do as to Ground B, which was the plasticizer business? If you will look at Page 5 of the decision, I think you will find it in the last paragraph. A. You want me to read it? What he says in the second sentence, he says, "A reading of the Heiss specification clearly indicates that the plasticizer need not be present when the polyurethane adduct is actually formed. The use of a plasticizer is considered not critical."

In other words, he is saying, in effect, that Heiss is entitled to claims which are not limited to a (713) plasti-

cizer.

Mr. Sweeney: Mr. Clerk, can I have Defendant's Exhibits K and L.

- Q. Mr. Upchurch, I will hand you Defendant's Exhibits K and L and can you just very briefly tell the Court what those are? A. Well, "K" is a petition by Price to the Patent Office asking the Examiner to reconsider and modify his decision on the motion to dissolve.
- Q. What is Defendant's Exhibit L? A. That is the decision on the petition for reconsideration.
- Q. What was the decision? A. He says, "Reconsideration of the Examiner's decision has been given but no change is made thereto."
- Q. Look, if you will, at the last paragraph in the decision on the petition for reconsideration. What is the Examiner doing there? A. Really the last paragraph says there is no appeal.
- Q. I mean the next to the last paragraph. A. The next to the last paragraph, the Examiner says he has considered the Windemuth patent 2,948,691, which shows

condensates of ethylene oxide and trimethyleipropane or pentaerythritol reacted with a poly organic isocyanate.

- (714) Mr. Sweeney: Now I will ask Mr. Clerk for Plaintiff's Exhibit 27.
- Q. Mr. Upchurch, I hand you Plaintiff's Exhibit 27 and ask you to look at the second part of it. The first part being a copy of the preliminary statement of Mr. Heiss, but the second part is what? A. This is the decision of the Board of Patent Interferences at the Patent Office. It is the Appeal Board or the board that decides an interference.
- Q. What was the decision of the board, if you would just explain it to the Court? A. Well, in essence the board decided that Heiss had no clear basis in his application for the molecular weight limitation of 600 and that he couldn't prevail in the proceeding.

On that basis they awarded priority of invention to Mr. Price.

- Q. Did they indicate anything about the Heiss patent or application being broader than the Price patent? If you will look at Page 13 of the decision, I think it might help you. It is up at top in the right-hand corner. A. They say that the Heiss claims 82 and 81 are broader than the counts, that is the claims that were in the interference.
- (715) Does that answer your question?
- Q. I believe that the next sentence would also be helpful. What does that mean? A. It says that it has been held that the fact that a broad claim embraces the subject matter of a narrower one does not in itself establish that the two are drawn to the same invention.
- Q. What did the Patent Office mean by that? A. Well, it meant that a claim—a broader claim does not necessarily establish that that invention and an invention in a more limited claim were the same.

- Q. The Heiss claims were broader? A. The Heiss claims were broader because they did not include the 600 molecular weight limitation.
- Q. To your knowledge did General ever raise their motion to dissolve on the ground of the Aroclor again? A. Not that I know of, no. Not in the Patent Office.
- Q. Were you active throughout the Price-Heiss interference?

The Court: Excuse me, would you read that question back?

(Question read.)

The Court: Are you now talking about the plaintiff in this action?

(716) Mr. Sweeney: That is right, your Honor.

The Witness: I answered that question thinking he said Price.

Mr. Sweeney: I should have said plaintiff. At that time it was not the plaintiff, it was General Tire in the Patent Office, and that is the reason I guess I got confused.

- Q. Were you active throughout the interference? A. Yes.
 - Q. Did you attend the final hearing? A. Yes.
- Q. Who represented Price and General Tire during this interference? A. Teddy TeGrotenhuis was primarily responsible. Frank Ernhardt usually attended and I think maybe attended all of the hearings.
 - Q. Who made the final argument? A. TeGrotenhuis.
- Q. Did Mr. TeGrotenhuis say anything about the priority of Price's invention at the final hearing? A. Well, when he commenced his argument he advised the board that he was no longer urging a prior reduction to practice by Price because he didn't have any records available to substantiate the contention.

Mr. Sweeney: Thank you. I have no further questions (717) of this witness, your Honor.

The Court: All right.

Mr. Kelton, Mr. Blecker and Mr. Kosinski, whichever?

Mr. Kelton: Yes, as soon as Mr. Sweeney clears, I will be glad to.

Mr. Sweeney: I did a little housekeeping, that is all.

Cross Examination by Mr. Kelton:

Q. Mr. Upchurch, when was this hearing before the Board of Patent Interferences that you last referred to? A. I would have to see the document. I don't remember the date.

Q. Don't you recall approximately when it was? Was it a year ago, ten years ago? A. It was several years ago.

Q. Several? A. Yes.

Q. Is that your recollection? How many years do you include in "several"? A. Well, I would guess or I believe it was some time between '60 and '65.

(718) Q. You have it bracketed. That is a little more than several, isn't it, years ago? A. I think it might be a little less than several.

Q. Well, we are dealing with the English language, Mr. Upchurch, and if you have a different definition for a word than I do, I have got to pursue it and see what your definition is. That is all it amounts to.

The board decision that you have just been reading from was mailed from the Patent Office on March 23,

1962, wasn't it? A. Yes, sir.

The Court: What exhibit are you referring to? Mr. Kelton: This is Plaintiff's Exhibit 27, your Honor.

The Court: I have it.

Mr. Kelton: And it is the third page in, the beginning of the board decision. That bears the stamp, your Honor, "Mailed March 23, 1962, U. S. Patent Office."

That means that is the date on which it was issued by the Patent Office.

The Court: All right.

Q. So this final hearing was somewhat prior to March 23, 1962, wasn't it? A. Yes.

(719) Q. Let me ask you: Do you remember anything more about Mr. TeGrotenhuis' statement to which you referred than that he said he was not pressing or not pursuing in substance the prior invention argument? A. Well, I remember that his statement included reference to a fire. I don't remember now whether it was at Notre Dame or General Tire, but it is my recollection that he stated that the notebooks had been destroyed.

Q. What do you mean by destroyed? A. Destroyed in the fire.

Q. Pardon? A. Destroyed in a fire.

Q. Did you seek out those notebooks at the time? A. Of the hearing? The time of the final hearing?

Q. No, at the time of the depositions. Wasn't this all stated— A. There were depositions taken by General Tire and I cannot recall any of Mr. Price's notebooks being produced.

Q. The statement was they couldn't be found, wasn't it? A. Well, my recollection was—which statement, the

one at the final hearing?

Q. The one that Mr. TeGrotenhuis made and otherwise the testimony. (720) A. My recollection of the statement before the board was that he was not going to urge a prior reduction to practice, actual reduction to practice by Mr. Price because the notebooks had been destroyed,

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Clelle W. Upchurch, for Defendant. Cross

the records, not just notebooks, the records had been destroyed in a fire. That is my recollection.

(727) Cross Examination by Mr. Kelton (cont.):

- Q. With regard to knowledge of the Price patent, I think you have testified that you saw the OG. A. I saw the claim in the OG, yes.
- Q. What is the OG? A. It is a publication that is issued weekly on Tuesday by the patent office in which they—well, at that time they published a claim from each patent that had been granted on that Tuesday. All patents are granted on Tuesday, incidentally.
- Q. OG stands for official gazette, does it not? A. Yes.
- Q. That is by the patent office, issued by the patent office? A. Yes.
- Q. You would have seen within the official gazette, the OG, you would have seen this within a few days after it was (728) published, would you not? A. Yes, within a week or so, probably.
- Q. So when you saw this notice of this patent to Dr. Price in the official gazette, what did you do, if anything? A. Well, I began considering the possibility of copying the claims in to this application, the Heiss application. I don't recall all of the details. I must have discussed it with Dr. Hardy, the research director, and I would have discussed it with Mr. Heiss and also with Dr. Saunders.
- Q. In your deposition which you gave in New York on January 10 and 11, 1972, in answer to a question, "Do you recall what was your next act in reference to this patent when you observed its issuance in the gazette," you answered, "Probably ordered a copy of the patent." A. Well, that probably would have been the first step, yes.

Q. That's what I really asked you, what you next did. A. I am sorry. I misunderstood you.

Q. What did you do when you received a copy of the patent? A. I am sorry, I don't remember the detail you expect, apparently. I probably took it out of the en-

velone.

Q. Let me ask you whether you discussed the subject matter of the Price patent with Mr. Heiss prior to your (729) preparing, in draft or otherwise, papers to try to copy the claims? A. Well, I don't recall whether I talked to him alone or not. I did discuss it with him, I discussed it with Dr. Saunders and I would have discussed it with Dr. Hardy.

The Court: Dr. whom?

The Witness: Hardy. Dr. Hardy was the research director at-

The Court: I just didn't get the name.

Q. With regard to the Heiss patent, do you recall an occasion when Dr. Saunders indicated to you he was not the inventor of some portions of that disclosure?
A. Well, I remember that he did do it, yes. It was when we were discussing the claims that were to be in interference with the Price patent, Dr. Saunders advised me, "Well, now, I did not contribute to that invention."

Q. In your deposition given on January 10 and 11, 1972, in this action, in this matter you stated that you didn't—you said, "But I don't remember the conversation. All I remember is that Dr. Saunders told me, 'Look, I did not make any contribution to the invention of the claims that would be in interference. My contribution related to manufacture of the foam with these'—we called them strong alkaline catalysts at that time." (730). Do you have any better recollection today of what Dr. Saunders said to you on that? A. Not really. He said what I just said, that he did not contribute to the invention which was to be the subject matter of the

claims in the interference. He did tell me that his contribution was related to those strong catalysts, the alkaline catalysts, I believe, that are disclosed in the specification, to making foams using those catalysts.

(733) By Mr. Kelton:

- Q. Now would you turn to page 156. You find such remarks, don't you? A. Yes.
- Q. At the date of submission of this amendment I see on page 158 it is dated September 17, 1962, Pittsburgh, Pennsylvania. A. Yes.
- Q. Did you prepare the remarks as well as the amendment? A. Yes, sir.
- Q. That's your signature on page 158, is it not? A. Yes, sir.
- Q. At the time, Mr. Upchurch, of preparation, this amendment, including the remarks in it, namely "on or before September 17, 1962," can you tell me whether or not you had or were familiar with the Windemuth patent number 2948961, which is Plaintiff's Exhibit 5 here? A. It was cited in the office action of August 13, 1962.
- (734) Q. Did you have any familiarity with it beyond the fact that it was cited? A. I was familiar with the patent. The Windemuth patent, or the application, was prosecuted by Dick Hutz of Connolly and Hutz in Wilmington. The application belonged to Bayer. I frequently discussed the Windemuth application and the interference with DuPont, with Mr. Hutz.
- Q. So you were familiar with the Windemuth patent? A. Yes.
- Q. I ask you to put your attention on the sentence in the second paragraph of the remarks, "Windemuth, et al., discloses only ethylene oxide." By "Windemuth, et al." do you mean the Windemuth patent 2,948,691? A. Yes.

- Q. Were you at that time familiar with the term alkylene oxides that was used in the Windemuth patent? A. Yes, sir.
- Q. I think you said you had a chemical background in your education. A. Yes, sir.
- Q. Weren't you familiar with the fact that the term alkylene oxides includes as the two most prominent members ethylene oxide and propylene oxide? A. If you leave out "prominent members," I can say (735) that I knew that alkylene oxides embraced ethylene oxide, propylene oxide, butylene oxide, amylene oxide, and many more.
- Q. Do you know how much amylene oxide is made and sold in the United States? A. Probably very little.
- Q. Is there much ethylene oxide sold in the United States? A. I think there is quite a bit of ethylene oxide.
- Q. How about propylene oxide? A. Apparently quite a bit.
- Q. Now if we can go back for a minute, Mr. Upchurch, to your amendment that begins at page 152 of the file wrapper contents, I notice in—and particularly on page 156 under the heading "remarks"—I notice that there is an indication here that there was a recent interview with examiner. Do you recall that? A. Yes.
- Q. Did you set down in the remarks the substance of what was said by you at that interview? A. Yes.
- Q. Can you point out to me in the remarks where you indicate that any particular thing was said at the interview? A. You mean where I state in the amendment that this was said at the interview?
- (736) Q. In the amendment, at the interview it was—A. I don't see any statement to that effect.
- Q. Mr. Upchurch, it is a requirement of a rule of the patent office, isn't it, that in every case where reconsideration is requested in view of an interview with examiner, that a complete written statement of the reasons presented at the interview as warranting favorable ac-

tion must be filed by the applicant? A. First off, I am not requesting in this amendment reconsideration based on a recent interview. I am requesting reconsideration in view of the foregoing amendment and the following explanation. The rules says you must acknowledge an interview. I acknowledged the interview in the next sentence.

I then preceded to summarize the discussion we had at the interview in the following remarks, and I don't know of any rule that says you have to state specifically that this is what I said at the interview and this is what the examiner said.

Q. It involves rule 133 of the rules of practice? A. Yes.

(743) By Mr. Kelton:

Q. Here is another one. I show you Plaintiff's Exhibit 40 for identification and ask you if you can tell me what that is. A. This is a letter to Mr. Sutherland from me, dated September 13, 1963.

Q. Who is Mr. Sutherland? A. Mr. Sutherland was a patent attorney in St. Louis. He is now dead.

Q. Was he in private practice or was he employed by Monsanto or— A. Private practice.

Q. Private practice in St. Louis? A. Yes, sir.

Q. What was the purpose, if you recall, for your writing this letter to Mr. Sutherland? A. Well, apparently Mr. Mahoney had asked me about the patent and I frequently consulted Mr. Sutherland when I wanted to have his opinion, wanted to see—apparently wanted to see if he agreed with me.

Q. Who was Mr. Mahoney? I am not sure that I know. A. Mr. Mahoney was president of Mobay. Now, I (744) can't say that he was, on September 13, 1963. He was director of marketing in Mobay and then became president, but I don't remember the date.

Q. Either one of those positions? A. He was either director of marketing or president of the company.

Q. This letter, as you read it on September 13, 1963, was it intended by you to express your then views? A. Really, it's just asking for an opinion. It expresses what I knew at that time, yes.

Re-direct Examination by Mr. Sweeney:

Q. Mr. Upchurch, I think I had asked you on direct (745) whether or not you recall whether the plaintiff here, General Tire, during the Interference, raised that second ground in the motion to dissolve on final hearing, that part of the motion which related to the— A. I believe you asked me, yes.

Q. What was your answer to that? A. I don't recall Price raising the issue of that second ground.

Could I see it again, before I answer?

Q. Yes. I'll hand you a copy of the board's decision, which is Plaintiff's Exhibit 27, and I am also handing you Defendant's Exhibit M, which is the Price brief on final hearing and perhaps from those two documents you can answer the question. A. I would still like to see ground No. 2, which you are talking about.

Q. Ground No. 2, yes, sir.

I'll hand you Defendant's Exhibit I, which is the Price motion to dissolve, which contains ground No. 2. A. Without me reading more, assuming that ground B only relates to the ground that Heiss could not—or that the counts were not patentable to Heiss because of the fact that Heiss shows that the plasticizer was necessary or an essential requirement, that was never raised again (746) as far as I recall.

Q. If you will look at the file history again, on page 156, and if you would read the second full paragraph that starts on page 156 and ends on 157.

Would you read it aloud? A. The one that starts "All of the claims"?

Q. Yes. A. "All of the claims except for allowed claims 81 and 82 have been cancelled and rewritten as claims 87 through 98. It is submitted that the new set of claims cannot be properly rejected as unpatentable over Windemuth, et al. All of the claims require that the alkylene oxide be propylene oxide. Windemuth, et al., disclosed only ethylene oxide.

"Since Windemuth, et al., does not disclose the polyether covered by the new set of claims, applicant is not merely substituting the Windemuth, et al., polyether for the one disclosed by Rinke, et al. It is submitted that no one reading the two patents together could conclude that one must use a polyoxypropylene ether instead of the polyethylene ether glycol of Windemuth, et al., in order to make a polyurethane of improved characteristics."

(747) Q. Referring to the interview which you acknowledge in the first paragraph on page 156, is the argument stated in the second paragraph that you have just read the argument that you gave to the examiner during the interview? A. Yes.

Mr. Sweeney: I have no further questions of this witness, your Honor.

Re-cross Examination by Mr. Kelton:

(749) Q. Reading or considering the Heiss patent, I take it it was not part of your preparation. A. I am unprepared, sir. I am not involved in this matter at all. I am strictly an independent party and I made no preparation, made no effort to refresh my memory.

(753) WILLIAM COLBURN, called as a witness by defendant, having first been duly sworn, testified as follows:

Direct Examination by Mr. Sweeney:

Q. Where do you reside, Dr. Colburn? A. I live in Highland Park, Illinois.

Q. Will you state your educational qualifications, please? A. I have a Bachelor of Science degree in chemistry from the University of Chicago in 1935. A Ph.D in chemistry from the University of Chicago in 1938. (754) I did six months of post-doctoral research at the University.

Q. Do you have any honors? A. Yes. I was elected to Phi Beta Kappa and Sigma Psi.

Q. How about scholarships and fellowships? A. Yes, I was awarded scholarships and fellowships all the way through.

Q. Do you belong to any professional societies? A. Yes. I belong to the American Chemical Society and I was a director of the Chicago section.

I am a Fellow of the American Institute of Chemists. A Fellow of the American Association for the Advancement of Science. A member of the American Society for Testing & Materials. The American Institute of Physics. The American Oil Chemists Society.

I think there are several others.

Q. Do you have any professional licenses? A. I am a licensed professional engineer in Illinois and a licensed stationary engineer in Chicago.

Q. Do you have any teaching experience? A. I was instructor in physical sciences at the Chicago City College for four years, and I taught part time, the name of the position was lecturer in physical sciences at (755) Roosevelt University in Chicago for five years.

Q. What is your industrial and professional experience? A. While teaching, I held another job at the

same time at the Deavitt Laboratories in Chicago, who were consulting chemists. I held that job for six years.

I went to work for Ninol Laboratories, who are manufacturers of surfactants as research director for two years. That was 1943 to 1945.

Then in 1945, I started my own business called Colburn Laboratories, Inc. That is a business known as Independent Laboratory.

Q. What position do you hold in that business? A. I am the owner and the president.

Q. Dr. Colburn, throughout the testimony in this case, there has been the use of a number of terms which I would like you to clarify for the record.

First of all, would you explain molecular weight? A. The molecular weight is a term, an arbitrary scale, in which each atom is assigned some weight. Here, we have been dealing with carbon, oxygen and hydrogen atoms, maybe some others, but arbitrarily carbon has been assigned a weight of 12. Oxygen, which actually weighs one-third more, as 16 and so forth.

So, if we had, say, a thousand carbon atoms in a box and they weigh a certain amount, we have a thousand (756) oxygen atoms in another box, those oxygen atoms would weigh one-third more because each atom weighs one-third more than a carbon atom.

So nobody can weigh one atom or one molecule, so the relations between them are known and one of them arbitrarily was assigned to the weight of one because in the beginning, it was thought that if we did it that way, they would all have whole number weights.

As we got accurate, we found it didn't work out, so they have odd numbers like 55.84, but in the early days of chemistry, they thought that particular thing had a weight of 56.

So, anyway, these different atoms actually do have different weights per atom.

Now, if we have a molecule, which is something made up of atoms, for instance, the ethylene oxide had oxygen atoms, hydrogen and carbon atoms, a certain number of each of those, if you would just add up the weights of those atoms, you have the weight of that molecule. That is called the molecular weight.

Q. Now, we have had polyether, polyester and hydrolosis. I wonder if you can cover those three. A. Well, I made some charts which would help, if I may step down.

(757) Q. Please do. A. I just made these in the hotel room freehand so they are rather crude, but this is all I had

Ether refers to a certain kind of a linkage between the atoms. Much of this was explained by Dr. Mark at the beginning, but I think there was so much in such concentrated form that at this stage in the trial, perhaps it will be easier to absorb.

Where you have a linkage between carbon atoms to oxygen to carbon, as I have in the bottom third of this chart, which I have marked Number 1, that combination is called an ether.

An ester is a different kind of combination without going into it, it is not important just what it is, but the linkage between the groups of carbon atoms is different than it is in an ether.

You notice in this thing which I have labeled polyether diol, we have a group of three carbon atoms, then an oxygen atom, then another group of three carbon atoms and they are joined together by an oxygen atom and this group where oxygen is used to join two groups of carbon atoms is an ether.

In this case, the compound was called a polyether because we have a number of different ethers all in one molecule.

(758) A number of different ether linkages all in one molecule.

If, instead of the ether connections between these groups of carbon atoms we had what is called an ester linkage, that is a different thing than just the single oxygen atom, then the molecule is called a polyester because it would have a large number of ester groups all in the one molecule.

The important point here is that the groups of carbon atoms, these groups of three in this case, are joined in an ether—by an ether linkage and in an ester, they are joined in an ester linkage.

The ester linkage is something which reacts with water, that is water reacts with the linkage chemically. This is different from just getting wet or just dissolving.

A chemical reaction is something that produces a new substance that wasn't there. For example, if we mix sugar and water together, the combination in a sense looks like a new substance. It is syrup instead of thin water, and it has a sweet taste instead of a neutral taste like the water, but it really is not a new substance.

The individual things, the water and the sugar, are still there. By a simple process of evaporating the water recovers them in their original states, and if you put in more sugar, it gets sweeter and so forth.

(759) The properties are just a matter of degree of difference between the things that are there. They are all still there.

If you have a chemical change, then something entirely new is formed. For instance, sodium is a metal. Chlorine is a green gas. If you put those two together, you have common table salt, which is a white crystal and which we can eat. The two things from which it was made are entirely different character, they are poisonous and so forth.

We have created a new material that wasn't there at all. That is a chemical change.

When the water reacts with an ester, the water chemically splits it apart, and creates two things that weren't there before, an alcohol and an acid. It is not important what they are, but the point is that the linkage between these groups of carbon atoms is split apart and we no longer have this long molecule broken up.

In that reaction where water is the agent that causes this is called hydrolysis. There has been talk here about polyester urethanes being unstable toward hydrolysis. These polyester urethanes are unsatisfactory in that respect. They are sensitive to water in the respect that the water attacks them and breaks them down and destroys them.

(760) I don't mean that they are sensitive to water in the sense in which that term has been used here, where something may easily get wet or it may swell up when water gets onto it.

In the case of the polyesters, they are literally destroyed by the water. You can't dry them out and recover them, so that is the difference between a polyester and a polyether and there you have the definition of the term hydrolysis.

- Q. Now, Doctor, would you deal with polyoxyalkylene and then propylene oxide polymers and polypropylene glycol. A. I might start out with the top part of this, which isn't a direct answer, but it will help me answer.
 - Q. That is chart No. 1? A. Chart No. 1.

Mr. Sweeney: Which I will eventually mark. Shall I mark it now, your Honor?

The Court: Yes.

Mr. Sweeney: That will be Defendant's Exhibit AJ.

(Defendant's Exhibit AJ was marked for identification.)

Q. Referring to Defendant's Exhibit AJ. A. This is the chart I have been talking about up to now.

(761) The top third gives a couple of examples of polyols. Now, the term "polyols" refers to the fact that there are several hydroxyls in the molecule. Of course, that is all clear up to now.

However, I think some witnesses have used the term to include a product which has two hydroxyls. Others referred to those which have more than two hydroxyls and used the term diol where there are only two. I am going to try to be consistent and use the term diol to mean the case where there are two hydroxyls in a molecule and polyol where there are three or more.

If we are talking about a reaction with disocyanate or polyisocyanate, we have to have at least two hydroxyls to make a polymer and if we do, we get what is known as a linear or non-cross linked polymer and if we have more than two, then it can cross-link through reaction with these hydroxyls.

There are other ways to cross-link, but in this case, we are concerned with cross-linking through the hydroxyl part of it and so I noted here on AJ one that the polyols will cross link, but the diols will not.

- Q. This will be defendant's Exhibit AK. A. I have a number 2 here, if I may, so I suppose I could call it AJ-2.
- Mr. Sweeney: That will be all right with me. (762) (Defendant's Exhibit AJ-2 was marked for identification.)

A. I am sorry, I have to go back to AJ-1 to answer your question. You asked me about polypropylene glycol, propylene oxide polymer and so forth.

Q. And polyoxyalkylene as a term? A. Yes. If we have a polyether that is often called a polyglycol. Whether there is logic to it or not, it is and since I am in the habit of using those words, I may use the word polyglycol or polyethergycol or polyalkylene oxide.

Polyalkylene oxide is almost a good word for this because if you take something like propylene oxide and put a lot of them together, hook them up end to end to make the polyether, you have this product, so if you say this is a polymer of propylene oxide, you are describing it properly.

So I may say propylene oxide polymer or ethylene

oxide polymer or polyglycol or polyether diol.

Polyether diol is what it is and what we have been talking about. This, of course, is also a diol and will not cross link

Incidentally, I have some words here in green, which are abbreviations for primary in the top and middle sections of this exhibit and SEC for secondary in the bottom and that refers to the type of hydroxyl that there is.

(763) There is a difference in the reactivity of different types of hydroxyls. The difference between the hydroxyls is in the carbon atom to which the hydroxyl is attached.

If that carbon atom is attached to two other carbon atoms, like it is here in the middle carbon of the glycerol, that hydroxyl has different properties than a hydroxyl attached to a carbon atom which is connected to only one other carbon atom.

So, the one that is attached to the middle carbon atom

of glycerine is a secondary hydroxyl.

The one attached to the two ends or to the four ends of pentaerythritol are primary hydroxyls.

The two that are on the ethylene glycol are primary hydroxyls.

The two on the end of polyethylene oxide are primary. The two that are on the end of polypropylene oxide are secondary. So there is a difference between the polypropylene diol and the polyethelene diol in that the hydroxyls are secondary in one case and primary in the

other case.

This does make a difference in their reactivity.

I think I have answered your question.

Q. All right. Now, there has been talk about polyother (764) polyols and polyether diols. Has that been explained? A. No. There is such a thing as a polyol, which is not a polyether like pentaerythritol, for example.

There is such a thing as a polyether, which is a diol

like polypropylene oxide.

If these are made to react with each other, rather than simply be mixed together, we get the propylene oxide connecting to the hydroxyls of the pentaerythritol combining into one new molecule. This is a chemical reaction producing a new substance, and that new substance now has a chain of ether linkages, in fact, four chains of ether linkages, and it also has hydroxyls on the ends so it is a polyether and a polyol at one and the same time.

That is a polyether polyol.

We might notice that a mixture of a polyether diol and a polyol, which is not a polyether, has two substances which are reactive to isocyanates. Either one alone would react with isocyanate.

The polyether diol makes linear polymers. The polyol,

pentaerythritol, makes cross linked polymers.

If you had a mixture, just the physical mixture of the two reacting with a disocyanate, each could go its own way.

The diol could react with isocyanate, making a (765) linear polymer. The polyol could react with diisocyanate and make a cross linked polymer and you would have a mixture of the two polymers all in the same pot.

Q. What are the alcohols in those compounds, Dr. Colburn? A. Well, in the polyol, which I mentioned on the other side, this is the not polyether polyol, pentaery-thritol, we have only primary hydroxyls.

In the polypropylene oxide, which I indicated as polyother diol in the top half of AJ-2, we have only secondary hydroxyls.

In the reaction product the thing that has been referred to in this case as a condensation product of propylene oxide and pentaerythritol, we would have secondary hydroxyls.

I don't want to give the impression that polypropylene oxide or this condensation product is exclusively secondary, but it is primarily secondary. The reaction to produce these other linkages and these hydroxyls can go either way. They can appear on the end or in the middle, but, in fact, they go very predominantly in the middle. So that this secondary structure represents basically what the product is.

Q. There was testimony during plaintiff's case about the (766) combination of the Hill Patent, which is Plaintiff's Exhibit 9, the Sokol patent, which is Plaintiff's Exhibit 6, and the Bayer article, which is Plaintiff's Exhibit 7.

What was the process that Dr. Mark indicated was obvious from those three references and if you would use your chart so you can explain it? A. The testimony was, as I recall, that the Bayer article shows cross linking with urethanes and, in fact, cross linking is very old. It was discovered by Goodyear in the case of rubber by Vulcanizing rubber with sulphur.

Dr. Mark explained very well the benefits of this kind of a reaction.

Then he pointed out that the principle of cross linking is shown, the benefit, that one would expect to get from it and, therefore, we have been taught that cross linking as a principle was known before the invention in dispute here was made and that feature of it, then, is old.

The principle is known. The differences between the thousands of patents that have been issued that incude cross linking are the differences in the materials

that are used to make the cross links and the materials that are cross linked. If the establishment of this principle were enough to make obvious any cross linking thereafter, there are literally thousands of patents that would have to be (767) deleted from the records.

That alone doesn't make it obvious to use a particular material, to cross link a material and it also doesn't teach how to cross link that material. These various inventions reside not in cross linking, but in the material selected to be cross linked and in the manner in which the cross linking is done.

The Hill Patent teaches the reaction of polyether diols,

including polypropylene diol, with diisocyanate.

In other words, this teaches the reaction of a material like the top right of Chart AJ-2 with disocyanate and this makes a linear polymer which is not cross-linked, as far as that part of the reaction goes.

Then Hill goes on to cross link by another reaction. He has a large excess of diisocyanate and he adds water and that creates another reaction, which creates the cross links, and he makes a kind of rubber.

So Hill was aware of the need to cross link. It was his purpose to cross link, but the way he did it is not the way that is taught in the Heiss Patent.

In other words, there is no suggestion whatever in Hill that cross links be made by creating some kind of a triol and using that to arrive at the cross links.

He has the polyether diol. He is reacting it with diisocyanate. He is interested in cross linking, but he (768) doesn't do it this way and there is not the slightest suggestion that it should be done this way.

This question of obviousness is a difficult one for me all the time because, as I understand it, the question is whether it would have been obvious at the time to a person having ordinary skill in that art. To put myself back into the framework of that time might not be

too difficult if I were familiar with the field at the time, but it would also require deleting the end product from my mind.

The thing is nice and obvious when we know what we are aiming for.

The Sokol patent teaches how to make a product like this one in the lower half of AJ-2. In other words, Sokol has taken the mixture on the top half of AJ-2, and used them to react chemically to produce the condensate that we are dealing with here.

There is no suggestion there at all that this condensate can be used to react with isocyanates to make something. Now, what we have in our invention is a condensate, like Sokol made, reacted with excess isocyanate so we get cross linking and producing a new product.

The question is was it obvious to take this product of Sokol, take the fact that Hill had reacted the diol with (769) urethane and produce the products of the invention in suit.

Now, when I put it this way, this all seems very simple and obvious. I have these two things laid out on the chart, I have the product laid out on the chart, and all you have to do is put them together. But if nobody had suggested, sokol had not suggested that this had any utility at all in any kind of a reaction with something where it is condensed or cross linked, he has a list of uses for it, but there is no suggestion of any kind of a reaction with anything resembling an isocyanate and Hill has no suggestion of cross linking.

Consider a worker who starts out to make some kind of a rubber. To arrive at this simple thing that may look pretty clear to us now, he had to cull these out from a mass of literature.

The attorneys have culled these out from the mass of literature after they knew what they were looking for. In other words, they had the benefit of the hindsight to guide them to Hill and Sokol.

The Court: May I interrupt?

The Witness: Surely.

The Court: As I understand it, the Hill patent, your explanation is a reaction of diol polyol ether with diisocyanate?

The Witness: That is right.

(770) The Court: The Sokol patent is a reaction of pentaerythritol, which is not a polyether, with a polyether diol to produce the propylene oxide?

A. It is almost that. It is the reaction of the pentaerythritol with propylene oxide to produce this product that is on the bottom.

The Court: Is it the reaction of the pentaerythritol—the polyol which is not a polyether with a propylene oxide?

The Witness: Right, and other things, but this is in there. It is one of the things that is in there.

The Court: All right.

The Witness: What I am saying is that with the benefit of hindsight, we can pick out these references and say, here it is all laid out.

You go from this to this to this and you have it, but if you didn't have the end product, the disocyanate reaction product of this polyol before you and didn't even know you wanted to make it, and you look at the thousands of patents in polymer art, would you pick these out and be led to this result?

The other criterion that I use is: Is there any suggestion whatever in these references that they can be combined?

This, to me, is a more objective way of trying to (771) determine this question than having a chemist look at these and say, "Well, sure, it would have occurred to me to do it." That is difficult. You can find fifty percent on each side. I suppose, if you get a lot of chemists.

Q. Dr. Colburn, would you explain the differences in the properties of these polyether—

The Court: Isn't there one more patent?

Mr. Sweeney: There is and I am going to come into that.

The Court: I thought you asked him a question about three. He answered two.

Mr. Sweeney: I want to set up a little predicate.

A. He asked me about three, the Bayer article and these two patents and I think I have answered that.

The Court: All right.

Mr. Sweeney: The three I covered were Bayer article Sokol and Hill. There is one more, Windemuth, and I will be getting to that in just a moment.

Q. Dr. Colburn, would you explain the differences in the properties between the polyethylene and polypropylene materials that we have been dealing with here? A. The polyethylene oxide products, now I am talking about the condensate with the polyol and so forth, reacting with the isocyanate, so we have the finished product of the (772) type contemplated by the Heiss patent, but if one had used ethylene oxide instead of propylene oxide in the process of that patent, then the products are sensitive to water.

I don't mean they hydrolyze. The whole idea of a polyether, instead of a polyester, is an advance because we have climinated the attack of water on the product.

Now, we have done that, they are not destroyed by water, but water does something to the ethylene oxide products that it does not do to the propylene oxide products. The ethylene oxide products are hygroscopic. That is in a humid atmosphere, moisture is absorbed into them and they get damp, they swell up.

If you had a seat cushion made of that material in a humid atmosphere for quite awhile, and then you sat down on it, it would sink down and possibly squeeze out enough moisture to become quite uncomfortable.

The propylene oxide products are not subject to that. They have tremendous utility and they are the things that are commercially used now.

The ethylene oxide products will swell up when put in water. Even some of the solid ones. I am not talking just about foam, but if you had, say, a solid piece of rubbery-like material and you put it in a tub of water, after a while, it would swell up almost double its size, (773) depending on which product you had. The propylene oxide products are resistant to that.

So there is a property here of the propylene oxide products which is not obvious. In other words, it isn't just whether this chemical reaction was obvious to carry out that is involved in whether the invention is obvious in view of the prior art, it is also whether the products that are produced have some unexpected unobvious properties.

Now, there was testimony that this good water-resistant property of the polypropylene products would be expected and obvious because polypropylene glycol is insoluble in water whereas polyethylene glycol is soluble in water, so one could reason from that that the polymer made from a polypropylene glycol would be water-resistant. That may be pretty good reasoning.

The trouble is that polypropylene glycols of the type we are talking about here are water soluble. The reasoning was based on the premise that they are insoluble in water, but they actually are water-soluble, and I notice this from my own experience, but I looked it up in a standard reference called the Encyclopedia of Chemical Technology and I have made some Xerox copies of that.

This is a book edited by Kirk & Othmer. It is a standard 14 or 15 volume work published over a period of (774) years as the various volumes came out.

Volume 7, which has this information, was published in 1951, it happens, although for solubility of the material

doesn't change over the years. If it was soluble then, it is still soluble now.

Anyway, this article gives the water solubility of polypropylene glycols of different molecular weights. These different molecular weights are made by building up the chain so it is longer and longer.

If you add other propylene oxide units so that weight builds up to 150, you have polypropylene glycol of 150 molecular weight and so on.

The materials that Dr. Price was making in his examples had molecular weights for the condensate of 640 from the one test that was in their data.

So, we are talking about something in that range here. Polypropylene glycol with a molecular weight of 150 is completely mixable with water in all proportions. Whether you add one to the other or what ratios, you get a clear mixture, just like mixing alcohol and water.

At a molecular weight of 400, the same thing happens. At a molecular weight of 425, the same thing happens.

At a molecular weight of 425, the same thing happens. (775) At a molecular weight of 750, they are no longer completely mixable with each other. You can dissolve 15 percent in water, which is still a reasonably good water solubility. Maybe something like the way salt will dissolve in water.

As you go up from there, the water solubility does decrease.

The literature of the Dow Chemical Company doesn't have too many examples, but as far as it goes, it confirms this. They are manufacturers of the product.

Further on this, I took the bottle of material that Mr. Heiss had brought and the one which was a nine molecules reaction of propylene oxide with glycerol, I believe it was Defendant's Exhibit AF, has a molecular weight of 614.

So that is a condensate in the molecular weight range we are talking about and I tested that with water and found that was completely soluble in all proportions.

Then further, Sokol in his patent—this is Plaintiff's Exhibit 6—at Column 8, line 7 describes the properties and uses of these condensates that he made and he describes them as being water soluble.

So, all the polypropylene glycols themselves in the weight range we are dealing with and even the condensate product, after somebody made them, are water soluble. (776) From this, it is not obvious at all that the urethanes will be water-resistant when it is known that the urethanes made from ethylene oxides are not water resistant. We have ethylene oxide polymers which are water-sensitive and the polyethylene oxides polymers are water-soluable.

Now, we take the polypropylene oxide polymers, they, too, are water soluble. So it would be unexpected that the urethane polymers would be water-resistant.

So, for those reasons, I believe it is not obvious to combine these references and obtain the product of the Heiss patent and added to that the reason that the product has unexpected properties.

(777) Q. In connection with this, Dr. Colburn, would you comment on the Windemuth patent, which is Plaintiff's Exhibit 5, and Dr. Price's article, which is Defendant's Exhibit C? A. The Windemuth patent discloses—incidentally, I am not using that word like a patent lawyer, if it has a special meaning, I don't know what it is—it teaches, discloses, describes condensation products which have ethylene oxide, although I grant that it has the term alkylene oxides and normally to a chemist that term alkylene oxide includes ethylene oxide, propylene oxide, but the chemicals themselves are all with ethylene oxide.

Then the properties of these products are described. In the Windemuth patent, which is Plaintiff's Exhibit 5, at column 2, line 12, it says it is talking about in every case that T am quoting the reaction product with isocyanate, in other words, the finished polymer, not just the polyglycol itself.

In this point in the patent it says:

"Products derived from polyglycol ethers of high molecular weight, however, dissolve in water and organic solvents."

Further down in the same column 2 at line 32, "The plastics are of interest because of their swelling properties.

For instance, it is possible, according to the (778) invention, to prepare rubber elastic products which are completely indifferent in aliphatic hydrocarbons, however swell in contact with water."

At line 53 in this column is the talking about these and some are water soluable and some aren't and it says: "The soluable representatives of this class of compounds may be used for a great number of applications."

Then it says, "They are suitable, for instance, as thickening agents, assistants in textile industry and some other uses."

Those two that I have just mentioned, thickening agents and assistants in textile industry generally require water soluability.

Column 3. Line 8:

"The films or foils thus obtained may have paper-like or rubber elastic properties. They are distinguished by a remarkable swelling behavior."

I will skip the next sentence. Then it says:

"The capability of the new products of swelling in water can be utilized if counter stresses are required of support materials such as paper, fabrics and films of high polymeric plastics, which films or foils are applied in contact with water or atmospheric moisture."

Now, when he said, can be utilized as counter stresses, maybe I had better explain it.

(779) You have probably seen that a piece of paper as it dries out or it goes back in humid atmosphere tends to curl one way or the other instead of lying flat all the time. This is true of many materials that absorb or dry out with moisture.

He is putting a layer of material on the opposite side so, let's say moisture would make this piece of paper curl so it is convexly upward, you put some of this new material on the bottom, that would make it tend to curl downward and the two would neutralize each other.

He is talking about putting a counter stress on by putting on a layer of material because it swells when it gets wet, just like the paper does, so this sentence here means that it swells in water.

The next paragraph starting Line 27 at column 3 says:

"Isocyanate modified polyglycol ethers, especially those have a molecular weight higher than 2,000 are soluable in water."

Further down at Line 59, "All these materials" and I am stressing the word "all", "however are distinguished to a larger or smaller extent by a marked swelling capacity in water and other solvents."

Everything in this patent shows that he is dealing (780) with products which are affected by water so it appears that he never even had the propylene oxide product in hand. All his comments show ethylene oxide. The properties of these products that he is talking about in general, which may be he referred to in general as alkylene oxide products, really are the properties of the ethylene oxide products.

The polypropylene product itself is not disclosed.

Now, if you consider Price's comments about this patent, I don't know what the exhibit number of this article was—

Q. Defendant's Exhibit C. A. In this article at Page 133, he is talking about his own patent and how he de-

veloped his invention and so forth during this entire talk, and then here he says, in the last paragraph on page 133:

"Two other important patent developments relevant to the development of polypropylene oxide polyurethane rubber were an American patent, issued August 9, 1960, to Windemuth, Schnell and Bayer, now footnote 3 and so forth.

Now, the footnote 3 shows the patent number he is talking about is 2,948,691, which is the same as Plaintiff's Exhibit 5 that I have just been talking about.

Then Dr. Price goes on to say:

"This patent claims products and processes of condensation (781) of diisocyanates with polyalkylene ethers with at least two hydroxyl groups per molecule and of molecular weight above 500.

While the claims cover all polyalkylene oxides, the disclosure and chemicals mention only polyethylene oxides."

So he interpreted the disclosure, aside from the chemicals, in the same way that I have just done.

Then he went on to say:

"These German chemists were working in a laboratory which led the world in developing polyester polyurethanes. In a patent filed over two years after our conception and reduction to practice they had clearly failed to recognize the significant advantage of the propylene oxide unit over the ethylene oxide unit in decreasing water sensitivity and crystallization."

In other words, Dr. Price says that this Windemuth and Bayer patent does not show propylene oxide. So at that time he interpreted this patent the same way that I just did.

Q. Dr. Colburn, when Dr. Mark was on the stand he talked about ether oxygens and the side chains on some of these molecules as being inert. Would you explain

that? A. Well, he was describing the reaction of diols or some kind of a polyol, I have forgotten which, I think it was diols with urethane in general and explaining how the hydroxyl reacts with the isocyanate to produce the urethane.

(782) In that connection, of course, the rest of the molecule is inert. So I don't quarrel with that at all.

But if we consider the original work by Bayer where they did not have long chains of polyethers, but had something else in there, then we can consider the effect of the presence of these ethers. They are chemically inert in this reaction, that is true, but the physical properties of the products will be different.

Just because you have the hydroxyls on the ends and react with isocyanate doesn't give you the same product in every case regardless of what is in between the hydroxyls, so I just want to say that what is in between is important and here we are dealing with whether the in between part being propylene units or ethylene units is important.

So we can't ignore this matter here.

Q. What about the side chains? A. Now, the side chains are these carbon atoms sticking out to the side. When the propylene oxide condenses with itself to build up this long chain, the oxygen link is connected to the end carbon of the chain of 3 on one and from there to the middle carbon of chain of 3 on the other. This is the way the reaction goes.

It is not a case of the two ends hooking up in a straight line so that you have 3 carbon atoms in a row between (783) the oxygens. They hook up so that there are two between the oxygens, repeating units of twos and then the methyl group sticks up on the side.

When you have ethylene oxide you also have repeating units of two so it would appear at first glance that there is a similarity, almost the same thing, and my contention

is that this methyl group sticking out, which is called a side chain, does have a profound influence on the products.

This is the thing that makes the product insensitive to water.

This little thing that you would think doesn't do anything makes all the difference in the world between the successful product and the unsuccessful one and this marked effect was unexpected.

The side chains produce some other effects like ability to maintain flexibility down at the lower temperatures and so forth. But the main unexpected result is this remarkably different sensitivity to water.

Q. Would you look at the chemical engineering article, Plaintiff's Exhibit 10, Doctor? A. Yes.

Q. How does this fit into the obviousness picture, if you will, of the Heiss patent? A. This is the article that gave out the news that a (784) novel polyurethane had been introduced at Princeton Plastics Laboratories and it was made from castor oil and disocvanate.

Now, castor oil is a polyester.

It is not a polyether.

It is a triol, but it differs entirely from the polyethers that we are considering here since it is a polyester and in that sense it is another chemical of the kind of polyesters that were in the original Bayer article.

In other words, going from polyester to polyether was an advance in itself regardless of which kind of polyether it was, and this is another chemical of a polyester.

T think it was put in for the purpose of showing that cross linking can be achieved through three hydroxyl groups and T grant it does show that, but that was known before.

In other words, it is an additional reference which to me has the same import as the Bayer reference.

Q. Now, Dr. Colburn, will you look at the Price file history, which is Defendant's Exhibit D. The Hill patent was cited against Dr. Price's application.

Can you tell the Court what the argument Dr. Price used to overcome this rejection in the patent office?

Mr. Kelton: Mr. Sweeney, are you qualifying Dr. Colburn as an expert in patent prosecution?

(785) Mr. Sweeney: I am qualifying him as an expert to read what is in the file history, that is all. I think he can do that.

Mr. Kelton: I will grant he can read it, yes, sir. Mr. Sweency: That is all he will do and tell his opinion of it.

A. On Page 50 at the middle of the page in the section which amounts to "remarks" by the attorney, it says:

"The patent to Hill is directed to a elastomeric sponge material formed by, one, a reaction product of a polyalkylene ether Glycol and a diisocyanate with, two, from about 100 to 600 parts by weight of water per hundred parts of reaction product."

Then skipping a paragraph, which deals with another matter, the last paragraph on the page:

"As noted in the patent to Langerak the Hill and Rugg references have no suggestion of the present invention in which polyalkylene ether glycols are cross linked with a coupling agent such as organic diisocyanate with a small amount of a polyhydric alcohol. As noted in paragraph 3 the Examiner also notes the lack of disclosure of a polyhydric alcohol pointing out that the references are directed solely to polyakylene oxide polymers unmodified by small amounts of highly functional alcohols."

(786) Then on page 61 about in the second sentence of the third paragraph are the words:

"There is no suggestion of the use of a condensation product of an alkylene oxide and a polyhydric alcohols in Langerak, Rugg or Hill. The condensation products of alkylene oxides and polyhydric alcohols in which the polyhydric alcohols are used as chain starters for the polyether glycol polymer are not disclosed in the above patents."

Those arguments are the same as my arguments.

- Q. There has been a lot of testimony about solvents and plasticizers in connection with the Heiss patent. Dr. Colburn, do any of the other references cited by the plaintiff disclose the use of solvents or plasticizers in urethane reactions? A. I think I saw one someplace in the Bayer article.
- Q. All right, look at that first. A. My note says it is on page 8. Yes, the second paragraph above the formula, page 8. Excuse me. This is of the Pretzel translation. Whenever I say Bayer article, I am talking about the English translation of it.

The Court: What exhibit is that?
Mr. Sweeney: That is Plaintiff's Exhibit 7B.

A. The paragraph I am referring to says:

"If one mole of hexane diisocyanate and one mole of 1,4- butylene glycol are heated in a monochloro (787) benzene solution for about one hour, a polyurethane separates * * *" and so forth.

In other words, he has an instance of the reaction using monochlorobenzene as a solvent. It is not unusual in this business to use solvents and, incidentally, monochlorobenzene is a first cousin to Aroclor. Chemically it would be about as inert or unreactive as Aroclor. If monochlorobenzene does not take part in the reaction except to serve as a solvent, one could reason that the Aroclors could also be inert in this reaction.

Q. How about the Hill patent? A. In the Hill patent—

Q. Which is Plaintiff's Exhibit 9? A. In Column 3 at the top, right at the first line:

"Although not critical to the operability of this reaction, it is often helpful in preparing uniform blown elastomeric sponges to thin the glycol isocyanate reaction mixture with a small amount of selected solvent such as acetone or dioxane."

So here is an instance where the reaction with the isocyanate is carried out in a solvent with the recognition that it is no more than a solvent to thin the mixture.

In this case the solvents acetone and dioxane are volatile materials which could be made to evaporate off later (788) if one wanted to. Chlorobenzene, which Bayer used, is less volatile than acetone, but it, too could be driven off by heating without too much difficulty.

These are recognitions that the reaction can be carried out with or without solvent. It is well known at the time that there is such a thing as using a solvent; that it may be inert in the reaction.

Q. How about the Price patent, which is Defendant's Exhibit— A. This is Defendant's Exhibit A.

Q. That's right. A. Example 3, which deals with the reaction of a polyether polyol with the diisocyanate is carried out in alpha chloronaphthalene and this is recognized as being a solvent here.

It says, "The polymer was dissolved in alpha chloronaphthalene and the reaction carried out and you can tell from the rest of the specification that there is a recognition that the reaction is between the hydroxyls of the polyol and the isocyanates of the TDI, et cetera, and the chloronaphthalene is nothing but a solvent.

Incidentally, alpha chloronaphthalene is a liquid and in this process it is converted to a dark brown elastomer,

which, of course, is a solid, so the formation of the polymer (789) in this liquid causes it to jell or solidify.

Alpha chloronaphthalene boils at 263 degrees C, which is so high that it would be very difficult to get it out. In essence here it is a plasticizer as well as a solvent.

Q. Dr. Colburn, during his testimony Dr. Mark said that all the examples of the Heiss patent, except Example 3, were triols where Example 3 showed a diol. I wonder if you would look at Example 3 and explain whether or not you agree with that.

Mr. Kelton: May I have the question? (Read.)

A. Well, the answer is a qualified yes. I agree that diethanolamine is a diol in that it has two hydroxyl groups. As I recall, he or somebody reached the conclusion from this that it would form a linear polymer instead of a cross linked polymer and that is not true. Diethanolamine has another group in it which also reacts with isocyanates and, so, it has two hydroxyls and an amine group, therefore it is trifunctional in this reaction, the same as a triol.

In any case, literally it is a diol, not a triol, but functionally it is a cross linking agent just like the triols are.

Q. Now, will you look at column 3, Line 69, of the Heiss patent, which is Plaintiff's Exhibit 1, and address (790) yourself to the language "may be plasticized"?

What does that mean to you? A. This deals with the question of the presence of or absence of the plasticizer and whether it is necessary to have it and here the patent specification says:

"In either case the products are relatively brittle resins which may be plasticized and rendered more flexible by effecting their preparation in the presence of a non-reactive organic material," and so on.

The question is whether this may be gives one the option of either using or not using the plasticizer and the testimony was that—so far—that this does not give one any option; that this word "maybe" has to be interpreted the way the term may be or maybe used was used other places in the patent.

Go to someplace where the inventor has used the same words "maybe" elsewhere in the patent, see what they meant there, in order to interpret what he meant in this case

Well, I think that is maybe a bit logical, but where you see how the word is used elsewhere and how it is used here I just don't see any possibility of confusing them.

For example, at the top of column 4 the words "may be used" are in the second line. In this case a list of plasticizers is given and it says "as illustrative examples of (791) nonreactive organic liquids and solids, which may be used in the practice of this invention, there may be mentioned" and then it gives a long list.

This to me is plain English, there is no problem. It means that if one uses one of these non-reactive organic liquids, he may use one of those in this list. There is no confusion to me about it.

Now, going to Column 3, starting at line 10. The phrase "may be used" appears there. In the specification there was an explanation that a class of materials that can be used in preparing products is the class of amines and here it says:

"Examples of amines which may be used in the preparation of organic compounds containing urethane groups are" and then there is another long list.

To me that means that if one is going to use amines he may select it from this list. In the next paragraph there is exactly the same kind of a use of the term. If one elects to use a certain class of materials, he may select it from this list.

Column 2, starting at Line 44, there is another place where the phrase is used in the same way. Suitable isocyanates, which may be employed are and then it gives a long list again.

(792) Right there the phrase "may be employed" means that if you are going to use a member of that class, you may select it from the list. No ambiguity whatever.

Now, at the bottom of column 3 it is used in another way. This is not a case where we begin with the option of selecting something from a list and then say, we may select from this list," this is a case where the option is whether or not to use a plasticizer.

It says "in either case," meaning whether you are making cellular products or some other kind or bubble free productions," in either case the products are relatively brittle resins which may be plasticized and rendered more flexible.

"In other words, they may be or they may not be. I just can't see any imperative need to use the plasticizer based on this phraseology.

Q. Dr. Colburn, there has been some testimony about homologs. Would you explain what a homolog is? A. Yes, I think I can do this better if I make a little sketch up here. I will mark this sketch AJ3.

Now, the idea of homologue or homologs involves a series of compounds which will have some property that varies gradually in degree as one adds more and more carbon atoms.

For instance, if one has a material which has, let's (793) say, ten carbon atoms in a row and it is a material like vaseline, say, sort of a soft jelly like material and one adds another carbon atom to make 11, it gets a little bit stiffer. If one adds another carbon atom to make 12, it gets a little thicker yet.

Little by little, if you get up to 18 or 20 carbon atoms, the thing gradually becomes more and more like a hard

wax. This sort of thing may happen with chemical reaction properties and so forth.

So, where we go from one compound to another by adding simply a carbon atom with two hydrogens attached, sort of inserted in the molecule to make it bigger, in most cases it is a kind of inert type of change qualitatively speaking. It doesn't affect the qualities, the things the material can do. It just affects the degree of what it already does.

If we take ethyleneoxide and propylene oxide, which is why we are concerned with the matter here, those are homologs of each other. The structure I have drawn in the middle represents the arrangement of the atoms, what we call a structural formula, for ethylene oxide and I will mark that EO.

The propylene oxide may be shown with a structure like this. That is the formula of propylene oxide. I will mark that PO.

They differ by the propylene oxide having a carbon (794) with two hydrogens in addition to whatever is in the ethylene oxide. I left off something here.

The reactive part of the molecule in the reaction we are dealing with is the part where the oxygen is and the rest could be considered as not being involved in the reaction.

So these two are homologues of each other. There may be some change in reactivity, a little more than you usually get going from 10 to 11 to 12 because, actually, this is a 50 per cent increase. It isn't just like adding one thing when you already have 99.

One added to two might produce a fairly big change, but, anyway, this is what is meant by a homolog.

Q. Are polypropylene oxide and polyethylene oxide homologs? A. This becomes AJ 4. I will just put in the carbon atoms and leave out the hydrogens to do this faster. I will keep on using red like everybody else for the hydrogens on the OH.

Then we have the ethylene units linked by oxygens. This is polyethylene oxide.

Now, the polypropylene oxide has these methyl side chains and each one of those is CH₃, I probably should put hydrogens in in one case so the difference is apparent. I will just put it on one pair of carbon atoms. (795) I will just take it one unit where I put the hydrogen atoms. If you count them you will see that the propylene case in that unit has CH₂ more than the ethylene.

That is because the original propylene oxide and the ethylene oxide differed in that way. These are just a lot of ethylene oxides hooked together to make a polyethylene oxide.

A lot of propylene oxides hooked together to make the polypropylene oxide and each of the units differs by CH₂.

But now consider the molecule as a whole, there is a lot more than just one CH₂ difference. To be a homolog this second formula would have one methyl side chain and no more.

I want to make it clear that polypropylene oxide is not a homolog of polyethylene oxide.

The properties of polypropylene oxide are quit different from those of polyethylene oxide partly because it has many side chains.

If you condensed one mole of propylene oxide with 99 mols of ethylene oxide, making 100 altogether, and compared that with a molecule where you have a hundred ethylene oxide units in the chain, those two would be very, very similar in their properties and those would be homologs.

But these differ by more than just one little side chain. There is a whole string of them. Every unit has a CH₂ and so these are not homologs of each other.

(796) Q. Dr. Colburn, during his testimony Dr. Mark said that propylene oxide was mentioned indirectly in Windemuth because of the presence of epychlorhydrin. Would you explain whether or not you agree with that? A. This was in column 4 at line 13. I think the chemistry is complicated here. I will give my reasoning though and my conclusion is that I don't agree with him, but it says:

"The use of epychlorhydrin gives rise to terminal alkylene oxide group which may be used for further reactions."

That part of the explanation shows that the chlorine of epychlorhydrin is the thing that was reacted and leaves the alkylene oxide group of epychlorhydrin unreacted.

Then two sentences above it is talking about the isocyanate—pardon me, the first sentence of paragraph, starting line 6, says, "Furthermore, the isocyanate modified the polyglycol ethers are valuable intermediate products for a number of further reactions."

So the reactions of the finished products are being dealt with here.

"For instance, N,N¹ dialkyl aminoethanols may be chemically added without difficulties, thus forming basic polyglycol ethers. These basic polyglycol ethers may be rendered quaternary by means of halides whereby new classes of (797) compounds are made accessible. The use of epichlorhydrin, et cetera.

Now, epichlorhydrin is a substance which would render that basic polyglycol ether quaternary so he is talking about a reaction which is entirely different from the reaction of propylene oxide to form the condensation product or the reaction with the isocyanate. This is an entirely different reaction, it has nothing to do with the subject matter.

(798) It just happens that epichlorhydrine, technically, is a derivative of alkylene oxide. Lots of things are derivatives of each other and that may or may not be of significance.

In this case, it is not of any significance at all because the epichlorhydrine is not used at all in the same way that the alkylene oxides are used in this invention in general.

Q. Now, Dr. Colburn, will you look at the Price patent, which is Defendant's Exhibit A.

Would you explain which of the examples in that patent support the claims? A. The claims are all directed to a reaction with a di-or polyisocyanate with a condensation product of a polyhydric alcohol and an alkylene oxide, to put it generally.

They all have the polyhydric alcohol as a feature and those are things that have 3 to 6 primary hydroxyl groups.

Now, example one uses as the glycol UCON fluid 75H1400 and then it explains what that is, a polyglycol copolymer of 75 parts of ethylene oxide and 25 parts of propylene oxide having two terminal hydroxyl groups, et cetera.

That, everybody agrees, is a diol. So, the work (799) with that is not in support of the claims of the patent, which require at least three hydroxyls. This diol has only two.

The code letter H in the UCON name refers to the fact that it is based on ethylene oxide and the first number 75, is the proportion of the ethylene oxide as compared with propylene oxide. This is part of the code of the UCON Carbide Company.

Going down to example four, it uses 74H1400 and the testimony of Dr. Mark was that he suspected that was the same kind of thing as example one, but he wasn't sure, so he really couldn't answer as to whether that was a diol.

Considering that the code letter II means these are the diols, in this case, if you took it literally, it would mean there was 74 percent ethylene oxide. I suspect it is a misprint, but in any case, it is still a diol, so example four also deals with a diol.

Example two has no isocyanate at all. It is directed to making a polyester, so that is not in support of any of the claims because they all require reaction with isocyanate.

Those are claims one, two and four that I have discussed so far.

Example 5 uses pluronic, and this, everybody has agreed, is a diol. So, this example also does not support the claims.

(800) Incidentally, I just happened to notice in that same column 5 there is a table of results of some tests at the top and it shows the volume increase on soaking in water for a product as being 172 percent. That is the middle column, the fourth item in that table, refers to the product RM7B swelling in water to the extent of 172 percent.

That is the rubbery material made by the reaction of the UCON 74H1400 with TDI. In other words, this is a diol isocyanate reaction product in which 75 percent of the alkylene oxide units are ethylene oxide.

I want to point out that that confers water sensitivity; this is another example of a place where the literature shows water sensitivity when there is a lot of ethylene oxide present.

Going back to this question, we have only example 3 and that is the reaction product of propylene oxide with pentaerythritol. That would be expected to give a polyether tetrol and then, that is reacted with TDI, so if there is any example here that supports the claims, this is it. This is the only one.

(801) Q. Now, Dr. Colburn, will you look—

The Court: Why don't we take a ten minute break.

(A short recess was taken.)

By Mr. Sweeney:

Q. Now, Dr. Colburn, would you look at the Herbst reports, which are Plaintiff's Exhibits 18, 20, 22 and 24, and tell the Court what the experiments you find as the source of Example 3 of the Price patent? A. Example 3 of the Price patent has certain details, for example, it says:

"70 cc's of propylene oxide, 6.8 grams of pentaerythritol, about 1.5 grams of sodium placed in a pressure reactor"—

The Court: Where are you reading?

The Witness: I am reading Example 3 of the Price patent.

The Court: I have it.

A. (Continuing)"—and the temperature of 142 to 150 degrees C."

Then it says, "This formed a viscous brown polymer." The only place in all four of these Herbst reports that has a reaction like that is on Plaintiff's Exhibit 18, the very first item, where he says:

"6.8 grams of pentaerythritol, 70 cc's of (802) propylene oxide and about 1.5 grams of sodium were used."

He has two reactions where that was done. One a pressure reaction and one a reaction at room temperature. The example refers to the pressure reaction. So the reaction of the example is No. 1, right at the top of

Plaintiff's Exhibit 18 and there is no other example in Herbst's reports which have just those proportions.

There are different proportions of propylene oxide or different amounts of sodium and so forth. The only difference as the example says 142 to 150 degrees C and Herbst's report has 140 to 150 degrees C and if we accept that as meaning the same thing, then this example follows the process of this particular Herbst experiment.

Q. Which is shown in Plaintiff's Exhibit 18? A. That is right, yes.

Q. Would you deal with the reaction between the pentaerythritol and the propylene oxide, Dr. Colburn? What happened there? A. Yes. Now, it is described in the disclosure of the patent and obviously expected from these Herbst reports and the letter that Dr. Price wrote afterward to the patent attorney, that was Defendant's Exhibit B, that a reaction took place between these ingredients to produce a reaction product where propylene oxide polyether chains were attached (803) to each of the original four hydroxyls of the pentaerythritol. Just like the bottom picture of Exhibit AJ2. I had that up there before.

Now, the reason for my having said something about primary and secondary hydroxyls is that in this reaction with the propylene oxide or ethylene oxide, primary hydroxyls are much more reactive than secondary hydroxyls.

The hydroxyls on the pentaerythritol are active. The propylene oxide reacts with them to attach itself and start building up the chain. As soon as the first propylene oxide combines with the hydroxyl pentaerythritol, you have a little chain and that chain has a newly formed hydroxyl on it, but that is a secondary hydroxyl, less reactive.

The other three hydroxyls in the pentaerythritol are still active as compared with the newly formed one and so some more propylene oxide would tend to react with

the remaining unreacted pentaerythritol hydroxyls rather than tend to extend the chain and this reasoning is also given in Sokol's patent, which was directed to making these kinds of compounds.

So, the point of all this is that with a lot of extra propylene oxide around, 20 propylene oxide molecules for every one of pentaerythritol, the attack (804) of the propylene oxide on to the pentaerythritol would use up the pentaerythritol right away and you couldn't have any left over. When the reaction goes, it attaches to the four primary hydroxyls of the pentaerythritol and then the chains build up and if at all goes smoothly as expected, all these propylene oxides would build the chains up and you would come out with a big molecule having these four branches and the molecular weight would be on the average 1,296 from these amounts of reactants.

Actually, the molecular weights, I don't know just what they were because they didn't run the test on this particular batch, but where they did run molecular weights, they were 640 and reading the specification it appears that they expected to have molecular weights in the 600 range, maybe 1,000, something like that. I really have no way to know what the molecular weight in this particular case is, but to the extent that we can say this reaction is analogous to others where they ran the reaction in somewhat the same way, it would be in the 600 to 700 range.

This means that the reaction, at least to a great degree, did not go as it was expected to go. They got a molecular weight of about half of what was expected.

In other words, these polypropylenes didn't add (805) on and keep on building up the molecules. Something else happened.

Then they found on Plaintiff's Exhibit 18 in this reaction about .5 grams of unreacted pentaerythritol was recovered and no doubt if a half a gram precipitated out

of solution, the solution that was left was saturated with pentaerythritol, so there was some additional amount of unreacted pentaerythritol. I don't know how much, but the fact is that the experiment shows that there was

unreacted pentaerythritol left over.

Now, that is very, very difficult to understand. Well, it is almost unconceivable that you would have all four hydroxyls of some of the pentacrythritol without having any propylene oxide attach to it while the rest of the propylene oxide was used up some way. There is no mention of some other unreacted ingredient like propylene oxide, for instance, and he did mention that he recovered the pentacrythritol so presumably if something else was left over, that would have been mentioned.

So just taking it on its face there was unreacted pentaerythritol and this just doesn't make sense if we are

getting the product that they expected to get.

In another exhibit, which I think is Plaintiff's Exhibit 24, yes, page 2, there is a paragraph, "Sodium (806) as Catalyst," and this is talking about an atmospheric pressure reaction, so it isn't precisely the same as our pressure vessel reaction, but as Dr. Herbst described his tests, the pressure vessel was open at the beginning, obviously it had to be open for him to put his ingredients in. So at that stage he doesn't have a pressure reaction and so it is just like the reaction in the glass vessel at atmospheric pressure.

He cooled to 5 degrees and then added the sodium and then allowed to warm up to room temperature. Up to the point where he put in the sodium at least, the pressure reaction and this reaction are the same.

Now, I don't know whether he put the cover on this pressure bomb before it came up to room temperature or not, but at least at that stage while it is warming up there is no significant pressure buildup as he is coming up to room temperature, so the reactions that go

on in that pressure bomb are probably just like what he is describing here.

He goes on on page 2 to say, "The mixture gradually became more and more milky and at 18 to 20 degrees refluxing commenced.

That means there was a substance present that was boiling. Refluxing means it was boiling. The vapors (807) are being cooled by a condenser, which is cold overhead and then the condensed vapors drop back down, that is what refluxing means to a chemist.

In other words, there was present some substance that boils at 18 to 20 degrees. Dr. Herbst said he didn't know what it was.

He was asked what it was on Plaintiff's Exhibit 22 there is a Herbst report and on the first page of that under a section headed "Summary" are four items listed. The fourth one says, referring to what is asked in this progress report, I am just reading the typewritten part now, "Details of the preparation of PO, PET polymers of molecular ratio of 100 to 110 to 1 are given."

Then Dr. Price testified that he in his own writing added these little editorial comments and he added the fact that this item four refers to the sodium catalyzed reaction and then he wrote in "an unidentified by product gaseous at room temperature was formed in each case."

So Dr. Price also could not identify this 18 to 20 degree boiling point substance.

If the reaction went to produce this polyether tetrol, as they thought it did, this substance would not be produced. There was no explanation for this being (808) there.

Now, I don't know what happened there, but getting a molecular weight of half that expected, finding some unknown, unexplainable by product, finding unreacted pentaerythritol left over at the end suggests that the reaction did not go as expected and it is very possible that they did not even have this polyether tetrol. I don't know

whether they did or not; it doesn't look like they did, but at the very least there is a substantial doubt as to whether they did ever make that at that time.

- Q. Dr. Colburn, would you now look at Plaintiff's Exhibit 20, which is the second Herbst report, on page 2, under Section c. A. Yes.
- Q. What is described there? A. There is described the reaction between propylene oxide polymers and disocyanate compounds.
- Q. What is a propylene oxide polymer? A. That is something that I had explained before. That is polypropylene oxide. Propylene oxide reacting with itself to build up a chain of propylene oxide units, that would be a polyether diol, a material which does not cross link.

Now, in this exhibit we are talking about, the (809) propylene oxide itself was allowed to react with sodium without any pentaerythritol being present.

Dr. Herbst, as I recall, testified he is not sure that he ever did this. Maybe he thought the pentaerythritol was always there, but this is a whole section of the report with a heading "The Reaction Between Propylene Oxide Polymers and Diisocyanate Compounds."

It is obviously a study of what happens when the

pentaerythritol is left out and left out purposely.

Well, the sodium reacts with the propylene oxide and there were two tests. One for 20 minutes, one for a whole day and in one case it said it gives a brown solid polymer, that is the one after 20 minutes. In the other case it doesn't say what the material looked like.

Then either one of these reacted with diisocyanate produced a hard, brittle polymer. So they got hard brittle

polymers without any pentaerythritol present.

Then it said, if the same reaction is used dissolved in diethylphthalate, a hard rubbery material was obtained. All attempts to dissolve this rubbery material in organic solvents have failed.

That was interpreted as meaning that cross linking had occurred.

These materials, hard brittle polymers, hard (810) rubbery material in the presence of the solvent or plasticizer insoluable in solvents are the very criteria that were used in the other experiments to conclude that cross linking had occurred.

The testimony was that, sure, it cross linked, otherwise you wouldn't have gotten these hard brittle materials. The fact that the material was insoluble in solvents showed it had cross linked.

Here we have hard, brittle cross linked materials that were produced by this process where the sodium was put in, the propylene oxide was put in, the diisocyanate was put in. The thing that was left out was the polyol upon which this polymer was to have been built.

So the fact that they got these hard, brittle polymers or rubbery material with diethylphthalate is no evidence whatever that they had a reaction between the propylene oxide and the pentaerythritol.

They got the same products described in the same terms even with the pentaerythritol being absent. Therefore, I think again that there is a very, very strong doubt whether they ever got this pentaerythritol polypropylene product, the condensation product, the polyether tetrol.

Q. Does Herbst show an excess of the toluene disocyanate, which Dr. Price said was necessary for this (811) reaction? A. Herbst's reports here nowhere show the amount of TDI, the disocyanate.

On page 3 of Plaintiff's Exhibit 24 the reaction, supposedly the details of the reaction, between the PO, PET polymers and the disocyanates are given and it starts right out saying:

"Qualitative reactions were carried out."

That means not quantitative.

The materials were not weighed to quantities.

The next sentence says: "The procedure used was as follows:

"About four to five cc's" again an indefinite amount, "of the PO, PET polymer was placed in a test tube and a few drops of the diisocyanate compound added."

That again is not a measured amount.

Then it goes on and apparently they cautiously added more drops and more drops until the reaction was finished. There is no measurement described for the ratio of the TDI to the condensation product.

One would infer from this, if this describes the details of the reactions in general, that there never was a measurement. However, the notebook isn't here and Dr. Herbst testified that he did measure and in Example 3 (812) were 15 grams of TDI for every hundred grams of the condensate was used must have been something that came from some one of the lost experimental notes.

Q. When you say Example 3, you mean Example 3 of the Price patent? A. Yes, excuse me.

Q. Which is Defendant's Exhibit A? A. Right.

Q. Please proceed.

How much TD1 is listed in Example 3? A. In Example 3 there is 100 grams of polymer dissolved in alphachloronaphthalene and 15 grams of TDI were added.

Now, the question is:

Is that an excess? Dr. Price testified that, of course it is an excess. You had to have an excess, the reaction couldn't go without an excess. You wouldn't get these hard products without it and so forth, so he knows there was an excess. But he couldn't pin down the source of this 15 grams.

Now, if we take this 15 grams we can calculate whether it is an excess. There is no problem in calculating that out, but I would like to put down a little explanation on a chart giving the basis for this calculation because (813) it is obviously an important point.

Q. This would be AJ-5, Defendant's AJ-5? A. Right. Just to make this simple I am going to represent a polyol by a wiggly line to represent the chain the middle part of the molecule and put OH's on the ends in red. That is the expected product, whose existence I doubt, but I can't be absolutely sure it is not there, but I strongly doubt it.

Now, this is to react with a diisocyanate and I will represent that by, let's say, a circle with the diisocyanate portions as little blue hooks. The reaction is one where the blue hook connects with the red OH.

To get a complete reaction where you have an equivalent amount of the isocyanates there would have to be as many blue hooks as there are red OH's and excess means that you have more isocyanate molecules than are sufficient to react with all the OH's so to make this work out right you would have as a minimum two of these diisocyanates for every one of the condensation product in terms of number of molecules.

Now, since these molecules have weights and we know how much they weigh and we know how much material was weighed out, it is possible to calculate how many molecules of each one were there. There are proportions (814) and this is what I did.

I have to make an assumption as to what the molecular weight is in order to make the calculation or else I have to know the molecular weight.

In the case of the TDI the molecular weight is known, that is 174. The amount you need to get one hook is half of that, 87. I will call that the equivalent weight. That is the amount that is equivalent to one of the red OH's. In this thing they made we don't know what the molecular weight was unless we assume that the molecular weight of 640 that was found in other cases was the molecular weight of this material.

So let us say the molecular weight was 640. The proportion of that that corresponds to each of the OH's is one-fourth of it so the equivalent weight is 160.

In order to have enough isocyanate to react with this tetrol, for every 160 grams of the tetrol we have to have 87 grams of isocyanate and to have excess isocyanate we need more, more than the 87.

In other words, the ratio 87 to 160 is the minimum amount of isocyanate that we need and we actually need more than that to have excess.

Well, it turns out that if I calculate how much isocyanate would be needed for 100 grams, the amount (815) they used in Example 3 of the Price patent, it turns out that they would have needed 55 grams. They actually used only 15 so there was nowhere near an excess even though they said they got these hard, brittle cross linked polymers, which proved there was an excess, and if we accept the number as representing an actual experiment, there wasn't enough.

Whatever happened was something else.

Now, let's give them the benefit of the doubt on whether this tetrol was formed and say actually it was formed. All the propylene oxide reacted with the pentaerthritol as expected to give the tetrol that was required the molecular weight of that then would have been 1296.

That can be calculated theoretically by just adding up the weights of the propylene oxide and the pentaerthritol that are supposed to have combined.

For each of the four hydroxyls in that case, then, we would have a fourth of 1296 which is 324.

In that case, then, we would have to have 87 grams of isocyanate for each 324 grams of the tetrol. Now, this is granting that they really did have the tetrol there and that I am all wrong in doubting whether it was formed, they would still need this proportion of isocyanate (816) as a minimum. For every hundred grams of this material,

the amount of isocyanate needed would have worked out to be 27 grams. So the 15 was still insufficient.

In this case would be needed 55 grams of the TDI. In this case would be required 27 grams.

I wrote in blue to represent the isocyanate. Therefore, in that experiment described in Example 3 there was no excess of isocyanate at all and whatever happened was not due to the reaction between this polyol and an excess of the diisocyanate.

Mr. Gweeney: Your Honor, at this time could I offer Defendant's Exhibits AJ-1 through 5, which are Dr. Colburn's hand made charts. I will, as soon as possible, have copies reproduced for the plaintiff and delivered to them.

Mr. Kelton: No objection, your Honor.

The Court: They will be received.

(Defendant's Exhibits AJ-1 through 5 received in evidence.)

Mr. Sweeney: T have no further questions.

The Court: Let me—before you finish your direct—let me be sure that I understand the contentions on this between the two parties. I am directing this to Mr. Kelton, too.

(817) I now understand by the difference between the plaintiff's claims and the defendant's contentions in respect of the prior art as regard the Bayer, Windemuth, Hill patent, I think I understand.

What I am sure of is—I don't want too much comment about it, but I am raising it so that you can have Dr. Colburn comment on it, since it may not be clear to me, and Mr. Kelton will be in a position to rebut it—as I understand the contentions here is that Heiss patent is invalid because of prior art, which I think I now understand the contention and that, in any event, if there was no prior art that Dr. Price—I am probably using the

incorrect term—reduced to methodology whatever is claimed in Heiss before he did it, is that correct?

Mr. Kelton: That is my contention.

The Court: You are claiming it is not and the testimony of Dr. Colburn particularly in respect to AJ-5 is addressed to that?

Mr. Sweeney: Our position is that whatever Dr. Price did through his lab assistant in 1949 he did not make Mr. Heiss' invention.

The Court: All right, I just wanted to be sure that I understood that.

Mr. Sweeney: What Dr. Colburn has been testifying (818) to is why.

The Court: This is a new science to me and I want to be sure I understood your contentions.

Mr. Sweeney: Would you like me to ask Dr. Colburn any more questions to help you out?

The Court: No, I think if I have stated what the contentions are correctly and Mr. Kelton nodded and you agreed to it, I think I understand. All right.

Mr. Sweeney: Then I have no further questions. The Court: All right.

Mr. Kelton: Could I have about five minutes, your Honor?

The Court: Sure.

(824) Cross Examination by Mr. Kelton:

Q. Dr. Colburn, in a good deal of your testimony on direct you were referring to stability toward hydrolysis, if I have my notes right. A. That is right.

Q. You made many references to stability of hydrolysis, didn't you? A. No. I don't think so. Just one.

- Q. Just one? A. I pointed out that the polyethers have a stability to hydrolysis that is not possessed by polyesters. So contrasting—
- Q. That would be unexpected, didn't you take that position? A. Not at all.
 - Q. No? A. No, I didn't say that at all.
- Q. Let me ask you: Would it be expected that they would have more resistance to water or more stability to hydrolysis? A. Would polyethers have more stability to hydrolysis than polyesters?
 - Q. Yes. (825) A. The answer is yes.
 - Q. You would know that? A. Yes.
- Q. That would have been known at the time Mr. Heiss claims to have made his invention and filed his application, would it not? A. Yes.
- Q. You have read. I take it, the Heiss patent because you have testified with regard to it? A. You are right.
- Q. I will ask you: Does the Heiss patent anywhere make a comparison from the standpoint of water stability between polyesters and polyethers? A. Well, when I read it. I wasn't looking for that point and I don't have any note marked on it.
- Q. Let me ask you this, Dr. Colburn: You said that castor oil was a polyester, didn't you? My notes indicate that you did. A. Yes, I did.
 - Q. A polyester.

Did you note any example in the Heiss patent using castor oil? A. I think there was one, at least. Yes, the last couple of examples, as I recall.

- (826) Q. How about Example 9? A. Right.
 - Q. That is an ester triol, is it not? A. Yes.
- Q. But you say it is polyester—polyester would result, right? A. No, I said the castor oil is a polyester triol.
- Q. A polyester triol? A. That's right, that is what my testimony was.

Q. You weren't then distinguishing its uses for reaction with polyisocyanate, then, as being a polyester reaction? Were you or weren't you? A. No. The reaction with isocyanate is between the hydroxyls. The polyester is elsewhere in the molecule.

Q. I beg your pardon, Dr. Colburn, because I got the impression you were indicating by your testimony that the Plaintiff's exhibit that was the Princeton work, you remember, I don't immediately recall the number of it, Plaintiff's Exhibit 10, was really polyester work? A. Yes, I did say that. But apparently what I have to clear up is the difference between—

Q. Before you do that— A. Excuse me, I didn't finish answering the question before the last one. I stood up to explain it and then you (827) asked me another question.

Q. Go ahead. A. That had to do with the difference between the polyether and the polyester.

Q. I didn't ask you, did I, the difference between a polyether and a polyester? A. In essence you did.

Q. All right, if you want to give it, go ahead. A. The ester linkages are along the backbone somewhere between the hydroxyls.

The hydroxyls react with the isocyanate and the polyester portion is the portion between the hydroxyls.

In the case of castor oil, the part of the molecule between the hydroxyls that react with the isocyanate has ester groups in it.

In a polyether it does not. The reaction with the isocyanate and the hydroxyl is the same in either case. It doesn't make any difference what is in the middle as far as that reaction is concerned.

When you are all through and have this big polymer with urethane linkages, the backbone in the middle breaks up because water attacks those ester groups and that is the difference, and easter oil is one of the things that

has three ester groups in it along with the three hydroxyls (828) that are also in the molecule. The reaction with the isocyanate and the castor oil is between the three hydroxyls on the castor oil molecule and isocyanates, but the part in the middle has esters in it just like other polyesters.

Mr. Kelton: Thank you.

Q. Now, I am still not clear, Doctor, what your position was with regard to Plaintiff's Exhibit 10.

I show you a copy of Plaintiff's Exhibit 10. I perhaps didn't understand your testimony on direct with regard to it so would you tell me what it is? A. It was that easter oil is an ester thiol and its reaction with diisocyanates is like that of other polyester polyols with diisocyanates and in that sense it is no different basically from the reactions of polyesters with diisocyanates that were the subject of the earlier German work.

The earlier German work, as well as this one, does not teach one to use polyethers or polyether polyols.

- Q. Now, can you tell me if there is any difference in substance between the work that is shown in Plaintiff's Exhibit 10 and what is set forth in Example 9 of the Heiss patent at the bottom of column 5? A. No, there is no difference in, again, speaking in generalities. (829) Q. So Example 9 is substantially the same thing as this chemical engineering article that was published in April of 1950, is that right? A. I think you used the word "substantially" and that gives me a little trouble, but it is the same chemistry. The same chemical process is going on.
- Q. Now, with regard to Example 9 of the Heiss patent, I ask you: Does Mr. Heiss indicate that this is an undesirable product? A. No, sir.
- Q. As a matter of fact, he says it yields a flexible bubble-free resin, doesn't he? A. Those are his words.

Q. I think you referred in answer to my question about castor oil in the Heiss patent, you were looking or about to refer to some other examples.

Would you point them out? A. You asked me if castor oil was mentioned and I quickly noticed that Examples

24 and 25 have castor oil involved some place.

Q. Yes, 24 uses castor oil and 25, if I understand it correctly, used a hydrogenated castor oil. A. That is right.

- Q. Would a hydrogenated castor oil be an ester triol? (830) A. Yes, sir.
- Q. These, except for the use of a catalyst, that is, by "these" I mean Examples 24 and 25, except for the use of a catalyst are chemically the same as Example 9 of the Heiss patent, aren't they? A. Well, I would have to consider the proportions and how much excess isocyanate there is and things like that.

Q. Will you please make the consideration, then, if you have to? A. I think I can without wasting too much time figuring.

Q. That is very good. A. Basically the chemical reaction of the isocyanate with the hydroxyl is the same thing in any case.

Later on excess isocyanate may itself take part in cross-linking and produce the gas and so forth, and the extent to which that will occur will depend partly on how much excess there is.

So I think you are looking for an answer to the question of whether the basic chemistry between the isocyanate and the hydroxyl is the same in all these three examples and if that is your question, the answer is yes.

- Q. Doctor, it is true also, isn't it, that castor (831) oil has secondary hydroxyl groups? A. Yes, that is correct.
- Q. And it is still able to react chemically, is that not true? A. The distinction between primary and secondary

that I was making was in the reaction with propylene oxide.

In this case we are talking about the reaction of the hydroxyl with the isocyanates so it is a different reaction, but the answer is the secondary reacts here.

• Q. Dr. Colburn, looking at my notes it seems that you made a distinction with regard to polyethers between polyethylene polyethers and polypropylene polyethers.

Do you recall that? A. Yes.

Q. That, as I understood, had to do with water sensitivity? A. It had to do with the water sensitivity of the polyurethanes made in the two respective cases.

Q. Water sensitivity of the product, the polyurethane

product made of the two? A. That is right.

Q. With reference to the Heiss patent, do you find any such difference disclosed in the patent specification? A. No, I don't.

(832) Q. Dr. Colburn, the only possible comparison would be in Example 10, it seems to me, or Example 11, excuse me.

There is a comparison there between the products of ethylene and propylene oxide condensates, then reacted with polyisocyanate.

Have I stated that correctly? A. I don't think there is a comparison. There is simply a listing of four examples, four reactions, and a statement that they all produced flexible bubble-free resins.

There is a comparison in the effect of the molecular weight; in other words, one with more ethylene oxide as compared with the one with less ethylene oxide is more flexible.

The same applies with the pair of propylene oxides, but basically this is not a comparison thing. It is just a statement that four reactions were carried out.

Q. On this water sensitivity distinction to which you have referred several times so far as polyethylene polyethers and polypropylene polyethers are concerned, was

not disclosed to the public or the Patent Office in this specification, as I understand you? A. That is correct. (833) Q. Isn't it true, Dr. Colburn, that so far as the ability to react—strike that.

Isn't it true, Doctor, that the reactivity, the qualitative reactivity of ethylene oxide and propylene oxide are substantially the same? A. Toward what? In what reaction?

- Q. Generally speaking. A. Let's restrict it to reactions of the oxide portion of the molecule and the answer is yes.
 - Q. Yes? A. All right.
- Q. There may be some difference in the rate of reaction between the two, but it is a relatively small difference, isn't it? A. Well, the words "relatively small" are relative terms, I suppose, but in my interpretation I agree.
- Q. Do you have any idea what it is; what the difference is? A. There are lots of reactions. If we are talking about the reaction with, say, the primary alcohol pentaerythritol, there isn't much difference.
- Q. In a co-polymerization reaction between ethylene oxide and propylene oxide, what would be the relative reaction rates? Have you got any idea? (834) A. That is a different animal than just having propylene oxide or ethylene oxide take part in the same reaction because once the reaction has started, you have secondary and/or primary alcohols in the already polymerized part of this thing, but in that reaction, let's say polymerizing a mixture of the two together to make, let's say, a random co-polymer, the reactivity of the ethylene oxide predominates and you would have predominantly ethylenes toward the center of this molecule and predominantly propylenes toward the end.

I don't have that combination on my charts, but you have it on your charts.

Q. Doctor, what you have said about this extension would apply to these polyethers, wouldn't it, that after

the first reaction, for example, with pentaerythritol, the reacting portions would be otherwise? A. That is true, but what you and I mean by "otherwise" may be different.

When we take the pentaerythritol and it reacts with the first molecule of propylene oxide, that would block off one of the primary hydroxyls that was on the pentaerythritol and create in its place a secondary hydroxyl, which is further out on the chain.

The next molecule of propylene oxide would (835) statistically speaking, tend to go to the primary hydroxls much more predominantly than to that newly formed secondary.

By the way, this is pointed out in the Sokol reference.

Q. Now, tell me, would the same thing happen with glycerine? A. Yes, generally.

Q. So there is no distinction with tri or tetrahydroxy? A. Well, you would tend to react with the two primary hydroxyls of the glycerine rather than extending the chain on one of them.

It would be unlikely to have a glycerine with one long propylene oxide chain and the other two original hydroxls left unreacted when you put in some number of propylene oxide molecules.

Mr. Kelton: T have some more for Dr. Colburn and T wondered how you felt about that.

The Court: My feeling about it is if it, is at all possible to keep us on our schedule for Monday, if we could finish with Dr. Colburn today; I frankly would like for us to do that rather than have a break. That is my own feeling as long as the reporter can hold up.

(836) The problem with breaking now is it will take a long time because in terms of lunch and that sort of thing, I prefer to finish and leave for the day because I have to go upstairs and do something.

Mr. Kelton: I think the inability to find any place to eat around here is one factor today.

The Court: My feeling would be let's go on.

Mr. Kelton: I had understood that you had hoped it wouldn't go beyond two o'clock today.

The Court: I didn't, but I thought that by that time we would be through with this witness.

If we are not, let's go ahead and see how far we can go.

At least I will go until Mr. Odette cries for help. He is the only one I think I will consider today. I think all of the rest of us have the strength to carry on.

Mr. Kelton: Then we will just slug along.

The Court: I have some work to do upstairs and I would like when we leave here to concentrate on that if it is possible, so we will go until the reporter tells me.

How long do you think you will be?

Mr. Kelton: It is a little hard to predict. I would think perhaps an hour and a half to two hours.

(837) The Court: We probably won't be able to go that far. Let's go as far as we can. Let's finish as much as we can today.

By Mr. Kelton:

Q. Dr. Colburn, you used the word "statistically" in answer to a question a few minutes ago.

What do you mean by statistically? A. Well, it is something like saying you have a 50-50 chance of coming up heads or tails tossing coins and if you toss lots of times, statistically coming out near 50-50.

In other words, I wouldn't expect to get 50 times heads and 50 times tails out of one hundred tosses, but the chances of having it come out that way are very good.

When I used the term it was something like that. Say the reaction rate is 10, 20 or 50 times as great for one reaction as for a competing reaction, then I would expect the products to reflect that difference in the reaction rates.

I would expect the product to represent mostly that species which would be produced by the more probable reaction.

I am trying to avoid saying something is 100 per (838) cent one way or the other or that I can tell you exactly what percentage goes one way or another just because the probability has been measured.

There is always a 95 per cent chance that you will have the thing come out as predicted. In other words, I suppose I am trying to be overly careful.

Q. That doesn't mean that all the molecules are one way or all of them are another. Statistically means there are some in between, some over and some under, doesn't it? A. No, not exactly. All the molecules of propylene oxide are like each other. All the molecules of ethylene oxide are like each other.

I am talking about, say, reaction where you put both of these in the same pot to react competitively with something else that is in the pot. Just because one is more reactive than the other doesn't mean that a hundred percent of the product will represent that one.

That is what I meant.

Q. Where you don't have them competing directly with one another in the same pot, let us say, then this reaction rate—any difference in reaction rate would just be a matter of waiting a little longer or a little less long for completion of the reaction in the case of one or the (839) other, wouldn't it? A. Yes, if we are talking about a single reaction. I don't think there was any example of that in anything we have been talking about, though.

Q. Let me get back for a minute to this polyester matter. With regard to the Heiss patent, Doctor, I asked

you if there was any comparison of polyesters with polyethers, I think. A. Yes. I don't know without study.

- Q. Now, is there mention of the polyesters in the Heiss patent as a specification there? A. That word "polyester"?
- Q. Or things that are polyesters? A. In column 2, starting around line 22 or thereabouts, for six or seven lines, there are things that include polyesters anyway.
- Q. Since you have gone to column 2, the portion which immediately follows the bottom of column 1, where Heiss speaks about polyglycols (ether glycols) then it continues over as polyethylene glycols and other glycols, polypropylene glycols in line 4. A. Yes.
- Q. Then following on in column 2, lines 9 and 10, is that the place you were referring to, a reaction of a (840) polyglycol with a dibasic acid and an ester and again esters in line 13. A. I don't know if you are interested in the distinction between a diester and a polyester or in talking about whether the two ester groups are within the molecule or not.

If you want to, I will have to go through these and tell you what each one is.

Hydroxy esters such as an ester prepared from one mole of dibasic acid and two moles of a glycol or polyglycol is a diester.

A polyester prepared so that the molecular ratio of glycol or polyglycol to the dibasic acid is two and one, that says polyester there.

An ester prepared from one mole of a dibasic acid and two moles of a glycol or polyglycol, that is a diester.

An ester prepared from a hydroxy acid and a glycol or polyglycol so that the molecular ratio of the glycol or polyglycol to the hydroxy acid is between .5 and 1, that is a monoester.

An ester prepared from one mole of trihydroxy compound and one mole of a monobasic acid, such as the monoglyceride of eleostearic, that is a monoester.

(841) I think that is the end of this group of esters

in this place.

Q. With regard to the matter of the water sensitivity properties of polyesters as compared with polyethers, you were in court, I think, when Plaintiff's Exhibit 62 was introduced and Mr. Heiss said he did not disagree with anything in there.

Had you considered before your testimony here this morning the portion on page 1— A. I have never seen this before just now so I have not considered anything

in it.

Q. May I read this to you:

"By both rigid and flexible"—I am reading from the summary here.

"Both rigid and flexible foams were prepared from

diisocvanates and a variety of polyols.

"Those foams prepared from ethylene oxide—and propylene oxide—derived polyols were greatly weakened by water.

"Foams from hydrogenated castor oil and polybutylene

glycols were not affected greatly by water.

"All of these foams were weaker than the best dissocy-

anate-polyester foams of comparable density."

Now, Dr. Colburn, doesn't that constitute from (842) the practical standpoint a diversion from your testimony here today with regard to the difference between polyesters and polyethers? A. No, it does not and I will explain why.

The effect of water on polyesters, like castor oil, for example, is a long term slow reaction where the water

destroys the product.

In this case he is obviously talking about the effect of simply wetting the product with water.

If you wet a propylene oxide or ethylene oxide foam with water, to some degree it will be weakened in the sense that you can tear it apart or something of that sort. Its tensile strength may be decreased. At least he is talking about that sort of thing here.

Now, if you put a hydrogenated castor oil foam or polybutylene foam in water, that is much more hydrophobic than just getting wet than is a polypropylene or polyethylene based foam and it is not affected greatly by water, as he says.

In other words, it sheds water like grease does and the other two foams do not.

In that sense the castor oil foam is not greatly affected by water as compared with an alkylene oxide based foam, but the long term effect of water on the castor oil (843) or other polyester foam is completely destructive, whereas the polyether foams will stand up and not be destroyed.

So there are two different kinds of effects of water that are involved.

When I said polyether urethanes are not hydrolyzed, I was very careful to explain that hydrolysis is the chemical reaction that destroys the ester connections and that does not happen with polyether foams.

When I talked about water sensitivity, I was talking about what happens when the foam or the product is put in water, whether it dissolves, whether it swells and so forth.

It is the sensitivity to water that is the subject of this report, not the long time stability to hydrolysis.

Q. Dr. Colburn, this is a report of work which was done from April of 1953 to April of 1954. It just wasn't a single water test.

Does that have any effect on your view? A. You are telling me it wasn't a simple water test which I suppose you mean what I just described. I don't know if that

is a fact without reading all of it, but just looking at the summary and the conclusion, it appears to me that my interpretation of it was correct.

(844) If there are long term studies of the stability versus hydrolysis of these different things and the conclusion from those is that the polyethers break down faster than castor oil, then I must stand corrected, but I would be extremely surprised if that is what has happened here.

Q. I take it, Doctor, that your testimony in part here is based on theoretical considerations as, for example, when you said that polybutylene would be more—I don't know whether you used the term "hydrophobic" or not—but less amenable to water? A. No, it is based on very practical experience and tests. For example, I have used polybutylene based urethanes to make ski boots where the resistance to water and cold is very important.

I do a lot of work in evaluating the stability and utility of these kinds of things.

For example, insulating—rigid insulating foam must be stable towards hydrolysis by water because if the foam gets wet or breaks down, it loses its insulating value.

I test these kinds of things in my laboratory all the time.

Q. Doctor, in connection with AJ-1, I think it was, you indicated a difference in reactivity as between (845) polypropylene oxide and polyethylene oxides.

That as I recall was based on the difference between the reactivity of a primary and secondary group.

Let me ask you: Is any distinction made in the Heiss patent with regard to primary and secondary groups so far as their reactivity is concerned? A. Well, I haven't studied it for that either. I don't recall ever seeing the words primary and secondary, but I suppose if they are there, they are there whether I saw them or not.

It would manifest itself probably in some statement as to whether one kind of material reacts faster or more readily than another.

I don't know if something like that there even. I would have to look for that.

Q. You have, I take it, made some study of the Heiss patent in terms of whether it supported some of the views you took? A. I didn't look at it to see whether it supports my views. I looked at it to see what is there and in some cases what is not there.

It is a lot harder to remember what is not there than what is there.

In this case I was talking about a chemical (846) reaction in general, this case meaning this reference to AJ-1, and I did not consider at all whether Heiss agreed with that or not or whether he said anything about it.

Q. Let me ask you: In preparing for your testimony here, haven't you examined the Heiss patent carefully to see if it propounded or disclosed some of these things that you say are so very important? A. Yes, I examined them very carefully to see whether some of the things I talked about are important and I didn't look at all to see whether other things are.

T, of course, read the patent several times and if something were there and impressed itself on my memory, I would remember it, but certainly I cannot answer you as to whether a particular word or explanation is absent without looking at it again if I don't remember now whether it is there.

Q. You talked about a mixture of, I think it was, propylene oxide polyol and ethylene oxide polyol—strike that.

Can we turn to the Windemuth Patent, Doctor? That is Plaintiff's Exhibit 5. A. I have a copy.

Q. I think you started out your testimony by saying with regard to the Windemuth Patent that it discloses (847) ethylene oxide only and then you had some talk about polyalkylene oxides and I also recall that when you said discloses, you weren't talking like a patent lawyer.

Now, let me ask you what do you mean by discloses? A. I was careful to say—

Q. As you used the term. A. I was not talking like a patent lawyer because I don't know exactly—

Q. You said you sounded like it at the time. A. But I explained that I did not intend to mean it the way a patent lawyer does because I don't know what a patent lawyer means by that.

Q. All right. A. I said it discloses or describes or tells about, meaning these are synonymous.

Q. Makes available knowledge as to, makes available or calls attention to a thing would be disclosing it, wouldn't it, to a chemist? A. I don't know about calling attention. If something is written in a book, I don't say the books calls attention to it, but it is there.

Q. Let me ask you this:

As a chemist, if anybody told you that a certain thing could be done with alkylene oxides, would that limit (848) you to ethylene oxide in your thinking? A. If that is all he told me, it would not so limit it.

Q. Then if somebody said things can be done with alkylene oxides, for instance, ethylene oxide, would that limit you as a chemist to a realization that it could also be done with other alkylene oxides? A. In that case it would depend on what are the things that could be done. If they are things that can be done only with ethylene, then it would be limited.

If they are things that could be done with others, then it would not be so limited.

Q. This is carrying out a certain reaction and I tell you as a chemist that this reaction can be carried out employing alkylene oxides, for instance, ethylene oxide. A. What is the reaction?

Q. For my purposes, Doctor, it doesn't make any difference what the reaction is. I have told you this and that is what we are talking about, disclosure; I have told

you about it. A. Well, in the case of most reactions, at least, if you told me it went with ethylene oxide, I would know that it would go with propylene oxide.

(849) Q. I have told you—maybe you didn't understand my question, Dr. Colburn—I am supposing that in this instance I have told you that a reaction—and I describe what the reaction is—can be done employing alkylene oxides, for instance, ethylene oxide.

I don't want you to misapprehend the question. A. I think I understood that and I think I answered it.

- Q. Well, can you tell me, did you answer it without qualification? A. No. You spoke about a reaction without specifying what the reaction was and I cannot say that I know right now as we sit here every reaction of alklyene oxides, but the reactions that I can think of at the moment would go with propylene oxide, if they go with ethylene oxide.
- Q. This would have been apparent to you, having normal skill as a chemist, back at the time you got your Bachelor's degree and Ph.D degree, wouldn't it? A. Yes.
- Q. Now, you referred to alkylene oxides, that you didn't know all the reactions of alkylene oxides.

I don't know quite what you meant, but let me clarify my problem this way:

Isn't it true that the two prominent alkylene oxides (850) are ethylene oxide and propylene oxide? A. Commercially speaking as of now, yes.

- Q. And that was certainly commercially and practically speaking back in 1953, 1954, 1955? A. Yes, around that time.
- Q. Now, I think that leads me to this polyether book that you had, Polyethers, Part I, page 150, if you will just look at it.

There in the middle of the page the statement is made:

"Ethylene oxide and propylene oxide have received an overwhelming amount of attention in the field of polyether synthesis."

Do you agree with that statement? A. As of what date?

- Q. This was published in 1963 and it speaks about the past. I cannot tell you what date. A. I am not sure about the dates, but my feeling is, say, starting about 1950 or so, that would be true.
- Q. Again, will you turn to page 94 of that same book? There is a statement there about the middle of the page:

"Propylene oxide is the first homologue of ethylene oxide and is similar in its chemical reactivity (851) to ethylene oxide."

Do you disgaree with that? A. No, I think I have testified substantially as this sentence reads.

- Q. That was known at about the time or certainly by the time you got your Ph.D degree from the University of Chicago, wasn't it? A. Yes, sir.
- Q. Now, you read a portion of Defendant's Exhibit C. Doctor? A. Yes.
- Q. And you say that Dr. Price treats it in the same way as you had done, as I understood your testimony? A. Yes.
- Q. Now, do you perceive that there is any distinction between saying, on the one hand, Windemuth does not disclose propylene oxide and saying, on the other hand, as Dr. Price did in this quoted portion:

"The disclosure and examples" meaning Windemuth "mention only poly (ethylene oxide)."

The Court: May I have the page you are reading from, please? I have the article.

Mr. Kelton: This is page 133, your Honor.

The Court: I have it now. Thank you.

(852) A. I am not sure I got that question, but I think you asked me—

Mr. Kelton: I will ask the reporter to read it to you—

The Witness: I had heard it. I don't understand it completely.

A. As I understand the question, you are asking me if there is a difference between saying propylene oxide is not there and saying ethylene oxide is the only thing mentioned.

If that is the—

Q. I certainly am not.

Mr. Kelton: Will the reporter please re-read the question?
(Question read.)

A. Unless—

Q. Perhaps, maybe the reason you don't understand it, maybe I didn't ask the question quite right. I will ask you again.

Do you recall that you said in your direct testimony, you started out and I think you said Windemuth discloses only ethylene oxide.

Do you recall that? A. Something like that.

(853) Q. Was that what you meant? A. Well, maybe there is some problem with what I mean by the word "discloses."

What I mean is that propylene oxide is not mentioned specifically and that the descriptions of the products made outside of the examples are descriptions which would apply to ethylene oxide products and not to propylene oxide products.

The examples themselves mention ethylene oxide. So in that sense propylene oxide, as a constituent in this reaction we are talking about, is not there.

Perhaps I should not use the word disclose.

Q. When you used the word disclosed with regard to Windemuth, did you mean that a chemist reasonably

skilled in the art, reading Windemuth would not understand Windemuth indicated you could use propylene oxide as well as ethylene oxide? A. I would say that a chemist reading this would say it excludes propylene oxide because of the examples and because of the properties of the products that were produced.

Q. I will have to take you back, Doctor, to ask you

what alkylene oxides are.

Have you omitted consideration of the disclosure (854) in column 1, about line 22? A. No, I said the word alkylene oxide is there. I think I said it right away.

Q. What does alkylene oxide mean to you? A. Alkylene oxide includes ethylene oxide, propylene oxide and others.

I agree with that, too.

Now we go on to read the rest of the patent and what he did and it is plain that every bit of work that is described here was done with ethylene oxide only.

Then he went ahead and extended this work to include

in his claims something additional.

Q. Is it your position, Doctor, that a skilled chemist reading the Windemuth patent and its disclosure with regard to alkylene oxides, for instance, ethylene oxide, that if he used propylene oxide in the same way, after reading Windemuth, in the same way that Windemuth did, he should be entitled to a 17-year monopoly of that as an unobvious operation on his part? A. If he got unobvious, unexpected properties in the products, then I think he is entitled to it.

Q. Now, what unobvious, unexpected properties are

shown-excuse me.

Let me ask you this: It is your position that (855) to do something which would be apparent to a skilled chemist somehow becomes unobvious if it has properties that he had not expected? A. I don't know about that particular person, but if skilled chemists in general

would not have expected the properties and, as I understand the patent law, this becomes a patentable invention.

Q. Isn't it true that from Windemuth it was apparent to any skilled chemist to try propylene oxide in place of ethylene oxide? A. No, the thing that is apparent to any skilled chemist is that if he tried the chemical reaction, it would proceed, but it is not obvious that he ought to try it.

Windemuth is getting products which are very useful and he describes them and that is that.

He has got good products, there is no suggestion that there is anything wrong with them or that something better is needed.

Q. And if those unobvious properties that you talk about were obtained, they should be disclosed to the art by the fellow who obtains them, shouldn't they?

Mr. Sweeney: Your Honor, I think Mr. Kelton is arguing patent law with Dr. Colburn at this point.

(856) Mr. Kelton: I am not concerned with that, Mr. Sweeney.

Dr. Colburn has evidenced a great facility— The Court: I agree, I think Dr. Colburn is an

expert and this is cross examination.

Overruled.

Mr. Sweeney: He is not an expert patent lawyer, that is the only point I made, your Honor. The Court: He knows how to answer the question. If he can't answer it, he will say so.

(859) By Mr. Kelton:

Q. Dr. Colburn, I think we closed on Saturday when I was asking with regard to the Windemuth patent, which is Plaintiff's Exhibit 5.

Do you have a copy of that? A. Yes, I do.

Q. Would you tell me the title of the Windemuth patent? A. High Molecular Weight Polyether Polyure-thane Polymers.

Q. That title would, to a chemist, make this of interest with respect to polyurethane products, wouldn't it? A. Yes.

Q. Do you agree that Windemuth informs the chemist that, and now I'll read portions from the space in the Windemuth patent, column 1, lines 20 to 23:

"It is known in the art to produce polyglycolethers in one or more terminal hydroxyl groups by polymerizing alkylene (860) oxides, for instance, ethylene oxide, or by chemical addition of alkylene oxides to polyfunctional alcohols such as trimethylolpropane, pentaerythrite, etc."

Do you agree that that is information that the Windemuth patent gives the chemist? A. Well, obviously, you just read it, so it's there. Unless there is some question about whether it's meaningful to a chemist. If that is your question, the answer is yes.

Q. No, that wasn't my question.

In other words, as I understand your answer, it is that that does appear as I read it in the Windemuth patent. A. Yes, sir, it does.

Q. Now, I'll ask you whether that doesn't suggest to a chemist that he can use propylene oxide as well as ethylene oxide for addition to a poly-functional alcohol or polyhydric alcohol having 3 to 4 hydroxyl groups? A. Yes, that part of the patent, taken by itself, would so suggest.

Q. Is it also correct, from the introduction of the paragraph I read, namely, "It is known in the art to produce," that Windemuth states that that information was in the art prior to him? A. Yes, that's right.

Q. Doctor, I show you Plaintiff's Exhibit 6, which we (861) call here the Sokol patent, and I ask you whether

that is not such a prior art patent showing the reaction of propylene oxide and ethylene oxide with a polyhydric alcohol of either 3 or 4 hydroxyl groups, to form a condensate. A. Yes. This patent does show what you just described.

The Court: Let me be sure that I am clear on this, again.

Doctor, one produces a polyester urethane product by—it isn't a question of whether it's alkylene oxide or propylene oxide, but it is whether it is polymerized with an alcohol with two or three hydroxyl groups.

Is that what the distinction is?

The Witness: No. The polyester really has nothing to do with propylene oxides or ethylene oxides. Polyesters are old, known before this art that we are talking about.

The Court: What is the distinction between them, then? Is it the hydroxyl groups that makes the distinction?

The Witness: No. The polyester has hydroxyl groups, just like the polyether does.

The polyester in the portion between the hydroxyls is different.

The Court: How does that come about?

Apparently I haven't gotten that.

The concentration of ethylene oxide has been the (862) thing that I thought there was a difference between the use of propylene oxide and ethylene oxide that would make the distinction.

The Witness: Yes, but that has nothing to do with polyester. The propylene and the ethlene oxides make two varieties of polyethers and one of these polyethers. I say, is unexpectedly unaffected by water. The one made from the propylene.

The Court: Now, tell me again, just quickly, what is a polyester? What is the distinction?

The Witness: A polyester would be the reaction product of something like ethylene glycol or glycerine with what is known as an acid and, assuming for the moment that you don't care what the structure of the acid molecule is, that then makes a residue from the acid molecule connected with a bond like that, and then the glycol—I got it wrong—this should be the glycol part (indicating on a chart). This would be the acid part, and so on.

We have groups of carbon atoms, but they are connected by this kind of a thing, which I am encircling in red, which is an ester, and the polyethers in place of this red circle thing, there is only an oxygen atom in the polyether. Otherwise, they are essentially the same.

This ester thing is a weak spot that's broken down (863) by the water and, therefore, for many applications, the polyesters, when made into urethanes, are not suitable, where the polyethers are.

So, to go from the old polyesters to polyethers is an advance and then, there is the further advance of the type of polyether and that's what this case is about.

Mr. Kelton: I would just remark, your Honor, that I think these differences between polyesters and polyether, such as they are, are included in the charts of Dr. Marks, Plaintiff's Exhibits 4-A to L.

The Court: I apparently had a misapprehension. I thought that the distinction was between the ethylene oxide and the propylene oxide, making the difference.

All right.

Q. Dr. Colburn, with regard to the Sokol patent again, in column 1, lines 5 to 10, that patent discloses the chemical addition of ethylene oxide or propylene oxide to pentaerythritol, does it not? A. Yes, it does. No question about that.

Q. If we can come back to the Windemuth patent, doctor, Windemuth also informs the chemist, does he not, the following, which I will take with a few omissions from column 1, lines 31 to 39:

"In accordance with the present invention, we have (864) found that glycol ethers can be reacted with polyfunctional isocyanates. The reaction results in novel products which may be used for plastics, spongy materials and the like." A. I agree it says that, if that's what you are asking me.

Q. Are you disagreeing that this would be information to a chemist?

That was my question. A. Yes. It conveys information to me as a chemist.

- Q. Further with regard to Windemuth, and here I will quote you from column 1, lines 40 to 53, with some deletions— A. Excuse me, if you don't mind. I wish you would not delete hecause I have a hard time jumping over the part you delete. I don't know which part it's going to be.
- Q. I'll tell you when I make a deletion and pick up. A. Fine.
- Q. "In the above reaction—deletion—addition products of alkylene oxides may be used as polyglycol ethers. Examples of isocyanates are"—and then there is considerable deletion—"appropriate poly-functional isocyanates, such, for example, as hexamethylenediisocyanate, naphthalene-1,5, diisocyanate, tolylene diisocyanate"—and then I would insert in brackets and add the word "and" and close the bracket—"4-4'-diphenylmethane diisocyanate. Depending on the (865) polyglycol ether and the nature and quantity of the isocyanates used in the reaction, products are obtained which, as compared with the starting material, show modified properties and open new fields of application."

Do you agree that Windemuth gives that information to the chemist? A. Yes, sir.

Q. Let me ask you, isn't it true, Doctor, that these three parts of information in the Windemuth patent to which I have called your attention—that is, the portion from column 1, lines 20 to 23; column 1, lines 31 to 39; and column 1, lines 40 to 53, suggest to a chemist to react a condensate of propylene oxide and a polyhydric alcohol having 3 or 4 hydroxyl groups, and an organic polyisocyanate to produce a polyurethane polymer? A. No, they don't suggest specifically propylene oxide. These are general statements that talk about alkylene oxides in general, and that includes propylene oxide but it doesn't specifically suggest any one alkylene oxide.

One would have to read further to see if a particular one is suggested.

Q. Is it your position that in order to have a disclosure there must be a specific suggestion as distinguished from a generic suggestion which covers the two main species (866) in the genus? A. I don't know what the word "disclosure" means as you used it.

I would say that one has to read the whole patent to see what it says, and the part you read me was just the generalizations in the introductory part.

Q. Don't these three parts that I have read to you indicate to a chemist that he may, if he wishes, react polypropylene oxide and the plyhydric alcohol having 3 to 4 hydroxyl groups with an organic polyisocyanate to produce a polyurethane resin? A. Yes. If he wished to carry out this reaction, this generalization would suggest that that reaction would go.

Q. That gives him the idea, does it not? A. No, it doesn't give him the idea. It just tells him that it is known that alkylene oxides in general take part in a certain chemical reaction. They have the same chemical

property and if one of them reacts, the others will, but it doesn't give him the idea to use any particular one.

Q. At line 40, Doctor, there is the disclosure or statement by Windemuth as follows:

"In the above reaction, addition products of alkylene oxides may be used as polyglycol ethers."

Now, taking into consideration that the above reaction (867) referred to was the reaction with isocyanates, isn't that a teaching to the chemist that he may use alkylene oxide as the polyglycol ethers in this reaction? A. Yes, he may use alkylene oxides or other polymers, or those with two hydroxyls or those with more than two hydroxyls.

In other words, it's a general statement which includes the condensation products of alkylene oxides with polyhydric alcohols, among other things.

The Court: Mr. Kelton, when you get to a point that you have finished an idea, 7'll take a recess for the sentencing.

Can you do that now?

Mr. Kelton: Yes.

(Recess.)

By Mr. Kelton:

Q. Dr. Colburn, I am still on Windemuth, on a different subject.

With regard to the feature in the Heiss claims of having an excess of organic polyisocyanate over that required to react with the reactive hydrogen or hydroxyl groups that are in the condensate, isn't it true that the use of excess isocyanate, and excess poly-functional isocy-

anate, in such a reaction is taught in the Windemuth patent, column 2, lines (868) 59 to 62? A. Yes, that's

right.

Q. It is true, is it not, as to that matter of excess polyisocyanate, that the Heiss article, namely, that by Heiss, Saunders, Morris, Davis and Hardy, which is Plaintiff's Exhibit 55, in its first paragraph—in fact, in the first sentence—indicates that that feature of an excess isocyanate was old to Heiss; that is, it occurred before Heiss? A. Well, I haven't seen this article before. I can agree that the first sentence says that the use of an excess of disocyanate has been studied before, but what the rest of the article says about that, I don't know.

Q. It is in connection with Vulcollans, is it not? A. Yes.

Q. Now. Doctor, here in court on Saturday you presented a calculation as to whether or not there was an excess in example 3 of the Price patent which is Defendant's Exhibit A.

Do you recall that? A. Yes, I do.

Q. From that calculation you concluded that there was no excess of polyisocyanate over the hydroxyl groups present in the condensate in example 3 and the Price patent, did you not? (869) A. That's correct.

Q. In that calculation, Doctor, you took the amount of condensate used, which was 100 grams, and the amount of TDI used which was 15 grams, and then you made two assumptions as to the molecular weight of the condensate.

()ne assumption was that it had a molecular weight of 640. Another was that it had a molecular weight of 1296.

Now, is that all correct? Do I correctly understand it? A. Yes, that's correct.

Q. Doctor, were there any other assumptions or considerations involved in this calculation which you made?

A. There was the assumption that the product was a tetrol. I am talking about the product called the polymer in this example, the viscous brown polymer.

- Q. If I understand what you have said correctly, that means, does it not, that you assumed that the condensate was a tetrol? A. Yes, for the purpose of this calculation, I assumed it was a tetrol. I did not say that I knew it was a tetrol.
- Q. Were there any other assumptions or considerations involved? A. No, none that I can recall now.
- Q. Do you have a copy, Doctor, of the Bayer reference, Plaintiff's Exhibit 7-B, particularly the translation? (870) A. Yes, I do.
- Q. In your direct examination, you made some references to the Bayer article and what it showed, and there are a couple of additional places that I would like you to look at.

Would you turn to page 16 on Plaintiff's Exhibit 7-B, table II.

Now, doesn't this indicate the applicability for reaction with polyisocyanate of a polyol backbone containing an ether bond, and I call your attention to the ninth and tenth item in the glycol column. A. Was your question whether those contain an ether bond? I think that was your question, and the answer—

Mr. Kelton: I'll have to ask the reporter to read the question.

(Record read.)

- A. Well, these have an ether in them and I have been using the word polyol to mean something with more than two hydroxyls, so I would say these are each a monoetherdiol.
- Q. Doesn't this indicate, Doctor, that the presence of an ether hand in the backbone does not interfere with the reaction in the case of these monoetherdiols? A. Oh. yes. I don't think anybody ever said it does.

Q. Then, you made a distinction between monoethers and polyethers, and I ask you, isn't it true that the material opposite the fusion point of column 208-212 is a polyetherdiol? (871) A. Well, using my usage of the term "poly" it wouldn't be. Some people may refer to poly as including two. Anyway, this has two ether linkages in the molecule and two hydroxyls. It's a dietherdiol.

Q. That again would indicate, would it not, the applicability of that dietherdiol, which I call the polyetherpolyol, as a backbone for reaction with isocyanate. A. Well, I don't know what you mean by backbone but this sub-

stance will react with isocyanate.

Q. In your direct discussion of the Bayer reference, you didn't say anything as to whether or not Bayer discloses or suggests employment of triols or tetrols to achieve cross-linking or reticulated polyurethane, as Bayer calls it. A. I think I did say that it's there.

Q. You did? A. Yes.

- Q. I am sorry, Doctor. I just didn't recall it, if you did, but may I direct your attention to page 24 of the translation which is Plaintiff's Exhibit 7-B, and I call your attention to the fact that what I am going to read to you appears under the heading, "Reticulated Polyurethane," and that, as I understand it, is cross-linked polyurethane; isn't it? A. It's the word chosen by the translator and obviously (872) it means what I mean by cross-linked.
- Q. In the next sentence, I'll ask you whether it's not true that Bayer discloses and teaches the following:

"If, however, diisocyanates are reacted upon compounds with more than two reactive H-atoms, such as glycerine,"
—I'll skip some of the others—"tridimensional macromolecules are insoluble in all organic solvents as well as infusible."

Do you agree that that is a portion of Bayer's teaching to a chemist? A. Well, what you read is what it says here.

It is chemical language. I don't agree with everything stated here, in that he describes tridimensional macromolecules, which is another way of saying cross-linked polymers and, depending on the degree of cross-linking and the substances that go into them, they may or may not be insoluble in all solvents.

As they get more cross-linked or the chains are shorter, they go in that direction, so the ultimately most cross-linked member in the group would be insoluble and infusible, et cetera, but there are all degrees of that.

One of these on the list is a polyether.

Q. There is no question in your mind that glycerine is a trihydroxy compound, is there? (873) A. No.

Mr. Kelton: I have no more questions on cross examination.

The Court: Mr. Sweeney.

Mr. Sweeney: I have no questions.

Mr. Sweeney: Your Honor, I would like to put in portions of DR. SAUNDERS' deposition which was taken the 19th day of January, 1972, at Monsanto Company in St. Louis.

I have a list of the pages by tine numbers, if I may just give it to the reporter.

I gave a copy of this to Mr. Blecker Saturday afternoon.

The Court: All right.

(874) (Page 6, line 19—Page 10, line 21)

"Q. All right. Did there come a time when you conducted research work in the field of polyurethane products, which are reaction products of polyisocyanates with compounds containing active hydrogen groups? A. Yes.

"Q. About when, approximately, did your research activities extend into that area of polyurethane chemistry? A. Sometime in the neighborhood of 1949, 1950, 1951. I don't remember the exact time.

"Q. Now, in 1949 do you recall the general scope of your activities in connection with forming polyurethane

products? A. No.

- "Q. Do you recall the general nature of the intermediates that you utilized in connection with the formation of polyurethane products in 1949? A. I remember the isocyanates we were working with at that time. I don't remember the exact date on which we started making polyurethanes from it.
- "Q. Now, do you recall the nature of the active hydrogen containing compounds that you utilized as intermediates for reaction with isocyanates in 1949? A. I think I don't understand your question, or (875) else you don't understand me. I don't remember specifically that we made those in 1949. We made them in a period from about 1949 to 1951, I'll answer the question on that period of time. But I don't remember specifically for 1949.
- "Q. Well, okay. My question was broader. I was speaking about the initial period of your involvement in polyurethane chemistry. Now, would you please answer, having in mind the starting materials that you utilized for reaction with polyisocyanate to make polyurethane products from the period 1949 to 1959.

"Mr. Rogers: If you recall. "Mr. Kosinski: Oh, yes.

"A. Our group worked with a variety of materials,

polyethers, polyesters, castor oils, glycols.

"Q. Do you recall just in general terms the type of glycols that you utilized during that period, namely, the '49 to '51 period? A. Ethylene glycols, glycerin, others similar.

"Q. Do you recall the molecular weight ranges of the products that you utilized during that period? A. When you refer to glycols, do you mean specifically low molecular weight glycols such as ethylene glycols, or do you

mean polyhydroxy compounds?

"Q. I'm asking you this in terms of you said you (876) utilized the certain groups of glycols, and I'm asking if you utilized the certain groups of glycols, and I'm asking if you utilized this in a general sense to include the whole range of products which are normally characterized in the art as glycols. A. I used the term glycols to refer specifically to low molecular weight hydroxy compounds such as ethylene glycols.

"Q. Now, did you use any glycols of a higher molecular weight than that type of product? A. The polyethers and the polyesters perhaps are what you are referring to, and those did terminate in hydroxyl groups. So

you might be calling them glycols.

"Q. So we can include the polyether glycols within the group of polyurethane intermediates that you utilized during that period of 1949 through 1951, is that correct? A. Yes. Research conducted by the group. I was the group leader at that time and—

"Q. Now, from 1949 until prior to April, 1951, would you please tell us who were the members associated with your group at Monsanto? A. I don't remember the exact

people for those specific dates.

"Q. Well, are you able to answer that question if you

(877) were to broaden out the period? A. Yes.

"Q. How would you broaden out the period in a fashion which would enable you to give me an answer to my question as to group membership? A. What would you like to have as the middle of the time span? Do you want it 1951 plus or minus two years?

"Q. I'll tell you, maybe I can be a little more specific. Mr. Heiss has testified in these proceedings that he

joined Mohay and he joined your group approximately April, 1951. A. Okay.

- "Q. Now, with that in mind are you able to identify the members of your group prior to the date when Mr. Heiss joined your group, namely, prior to April, 1951? In other words, from '49 until April, 1951, are you able to identify the members of your research group at Monsanto? A. As I remember, one was Hector Flores and another one was Clark Welch. There may have been others, but I don't remember specifically.
- "Q. Now, you identified one research activity as being involved in the research work in connection with the synthesis of diisocyanates, and we also discussed that certain aspects of your activities in this period were (878) concerned with making polyurethanes. Now, are you able to just state generally what were any other specific research projects of your group from 1949 until Mr. Heiss commenced his employment in April, 1959? A. We also worked on other non-isocyanate products of interest to Monsanto.
- "Q. How about in the field of any specific research project in connection with the application of diisocyanates? A. As I remember it, we did not start the application part until Heiss came."

(Page 12, line 17—Page 12, line 24)

"Q. I see. Now, do you recall independently the approximate time that Mr. Heiss commenced his employment at Monsanto? A. About 1951.

"Q. About 1951. To what group was Mr. Heiss assigned when he commenced employment at Monsanto? A. Mine.

"Q. Did he report directly to you? A. Yes."

(Page 23, line 23-Page 25, line 7)

"Q. Did you closely follow the course of Mr. Heiss' (879) laboratory experiments? A. Yes.

"Q. Would you please describe the physical layout of the research facilities in which your group conducted work at Monsanto in the period 1951 through 1953? A. At about the first of that period we occupied two laboratories in a small building which had a total of six labs. Mr. Heiss and I were in one lab, one or two other people were in the other polyurethane lab.

"Q. Did you jointly share the same laboratory? A.

Yes.

"Q. And how long did this continue with that physical layout? A. I don't remember exactly how long. A matter of one or two years. Sometime about 1953, the end of the period you asked about, we moved to the other building which had more laboratory space. Initially we were in the same laboratory there, but after some additional period of time, perhaps a year, we were in different laboratories.

"Q. Now, during the period that you and Mr. Heiss occupied the same laboratory space, do you recall the names of other members of your research group? I'm talking now about '51 to '53. You know, the end of '53. A. Yes. I think in that period it included a (880) technician named Marjorie Morris, a boy who was either a co-op student or a technician named Billy Davis, Walter Wade Robinson, Paula Combs and Gene Bond. There may have been others I have forgotten.

"Q. Now, what were the general nature of the duties of these various people in your group during that period? A. Mr. Heiss was primarily on polyurethane applications. Miss Combs and Mrs. Morris worked largely with him. Gene Bond was primarily on preparation of isocyanates. I don't remember the specific assign-

ments of the others."

(Page 26, line 3-Page 26, line 13)

"Q. Do you recall whether or not Mr. Heiss conducted any investigations of reaction products of diisocyanates

for use or possible use as adhesive type materials? A. Yes.

"Q. Would you give us the details of your recollection of that aspect of Mr. Heiss' work? Now, I'm speaking now of the period 1951 to the middle of 1953. A. He was interested in finding good adhesives which could be prepared from polyurethanes. He investigated the use of isocyanates such as tolylene diiosocyanate and diphenyl methane diisocyanate, reacting these with compounds (881) such as castor oil, such as polyethers and polyesters and glycols."

(Page 40, line 17-Page 41, line 4)

"Q. You say that you got all the notebook pages; were the notebook pages of the various persons who worked in your group submitted to you on a periodic basis for your review? A. Duplicate copies were turned in. Not on an exact schedule, but as the author wanted to turn them in, as they were completed.

"Q. For what purpose were they turned in to you! A. It was a good way of keeping up with what was being done. I passed them on to the patent department, I believe, for filing.

"Q. After you reviewed them? A. Yes."

(Page 44, line 1-Page 44, line 14)

"Mr. Kosinski: Dr. Saunders, do you recall a project, a research project conducted by Mr. Heiss which involved or had as its aim the formation of dielectric products by reacting a diisocyanate, a polyol and Aroclor?

"Mr. Sweeney: Objection. You're leading the (882) witness.

"(Discussion off the record)

"Mr. Rogers: Would you read the question?" (Previous question was read by the Reporter)

"A. I recall the project I think you're referring to, the Aroclor probably was not actually a reactant, but more likely was a solvent or a plasticizer in the system.

"Q. Do you recall that project? A. Yes."

(Page 47, line 4-Page 48, line 4)

"Q. Dr. Saunders, I'll hand you a document which has been marked as Heiss Deposition Exhibit 10 and ask you if you received a copy of the document which appears within pages 2 through 5 of this Exhibit, on or about the date indicated. A. I'm sure that I saw the report before it issued as a monthly progress report, but I don't remember that I actually got copies of them after they issued.

"Q. Dr. Saunders, I hand you a note book which has been marked as Heiss Exhibit 11 and I ask you, as a preliminary to my next question, if you could please consider the entries which are set forth on page num-

bered 84531.

"Mr. Sweeney: I might say that these red marks (883) on these pages are not part of the Exhibit, I assume they are yours.

"Mr. Kosinski: They are not part of the Exhibit, they are on our copy of the Exhibit. They

were applied by someone in our office.

"A. All right.

"Q. Do you recall if you became aware on or about the date indicated of this experiment which is reported on page 84531 of the notebook marked Heiss Exhibit 11? A. In general, I certainly knew of the work which he was doing soon after he did it, and I read copies of all of his notebook pages. I don't have specific recall of reading that specific page."

(Page 84, line 1-Page 84, line 25)

"Q. Dr. Saunders, do you recall whether during the course of Mr. Heiss' work on the so-called Aroclor

solidification project, that he ever produced a practical polyurethane product by reacting a polyether triol with a polyisocyanate in the absence of a plasticizer?

"Mr. Sweeney: I object to the question, it calls for a conclusion. It's indefinite, particularly the word 'practical'.

"Mr. Kosinski: Any product.

(884) "A. With my own interpretation of what is practical, I would say yes.

"Q. Would you please describe the nature of that product? A. It could be practical in the sense that it would be a reduction to practice of an invention and could lead to a patent.

"Q. Would you please describe the specific product that formed the basis for your last answer? A. I don't recall the expert experimental details

don't recall the exact experimental details.

"Q. Well, tell us how much you do recall. A. He reacted isocyanates, TDI and MDI with polyether polyols, obtained polyurethanes from these with and without catalysts, was able to jell the Aroclor material, obtained essentially solid polymers which were quite good in their flame resistance."

(Page 85, line 19—Page 86, line 24)

"Q. No. I said in his work in connection with the Aroclor project, if he ever produced a practical product in which he reacted these two reactants in the absence of a plasticizer. A. Then that depends on whether an experiment which did not include Aroclor would be classed as a part (885) of the Aroclor project.

"Q. Do you know if he ever conducted such an experiment? A. In which he reacted an isocyanate with

a polyether without Aroclor?

"Q. That's correct. A. He did.

"Q. Are you able to state the details of your present recollection concerning the experiments which formed

the basis for your last answer? A. He worked on a series of polymers which we considered to be of potential interest for adhesives, coatings, and possibly for other applications as well, in which he reacted isocyanates such as TDI and MDI with polyethers, primarily using a so-called pre-polymer technique. And these were then cured successfully to films, to coatings, to adhesives, and we occasionally got foams from them.

"Q. All right. Now, let me just explore the polyether aspect of your last answer. Would you please describe, if you can recall, the specific nature of the polyether adducts which were utilized in connection with those experiments that you have just described? A. He used polyethylene glycols, ethylene oxide derived triols, polypropylene glycols, propylene oxide (886) derived triols and polybutene glycols."

(Page 98, line 10-Page 99, line 25)

"Q. Dr. Saunders, you have had quite a lot of experience in the urethane field, and I think it might be valuable to the Court that is going to hear this case eventually if you would give us a little history of the urethane business. As I understand it, we started out in Germany during the War, is that correct? first work was done in Germany before the War, I think, at the I. G. Farben laboratories, beginning in about 1937. The initial work was directed toward the preparation of polymers which might be suitable for fibers competitive with nylon. Those particular products were never real commercial successes, but that research led the people at I. G. Farben and later at Farbenfabriken Bayer to explore the use of isocyanates for a number of other purposes. During the war the most significance uses very likely were in the area of rigid foam and adhesives. which the Germans did make use of in production scale.

"Not long after the war was completed, I. G. Farben which had been broken into segments, one of which was Bayer, did continue the polyurethane research work or, more specifically, the people at Bayer continued the polyurethane (887) research work and developed a very good flexible foam system, elastomer system, coatings, and continued the work with adhesives and rigid Monsanto and DuPont had started isocyanate work in this country, DuPont having been first, based on their patents which have issued. But I think very little was done beyond laboratory scale until the late forties when the work which had been done by the people in Germany became rather well known over here. It led to commercial interest in this country, first in the area of rigid foams, but the real breakthrough as far as this country was concerned was the flexible foam development, and that is still the largest application in this country. It has grown to the point that estimates, I think, indicated there might be a total of about one billion pounds of polyurethanes consumed in this country in 1971.

"Q. Now, can you inject into this history the difference between polyester foam and polyether foam? A. Do you prefer to limit that to flexible foams or do you want it to include both flexible and rigid foams?"

(Page 100, line 7—Page 102, line 21)

"Mr. Sweeney: The urethane work in Germany started with polyesters, is that correct?

"A. Yes, that's right.

(888) "Q. And then we have this other class of urethane materials which are called polyethers? A. May I back up just a little bit?

"Q. All right. A. The very first German experiments were utilizing—the very first experiments in Germany utilized hexamethylene diisocyanate and 1,4 butane diol

as reactants to make their candidate for synthetic fiber use. That was not successful. The first successful uses did employ polyesters as reactants with diisocyanates. It was polyesters that the Germans used in their rigid foams during the Second World War and also in their adhesives. Could I go on now to your question?

"Q. Yes, sure. A. Would you repeat for me his last

question?

"Q. Maybe I can just say it again. What I'd like you to do was to tell us about the difference in this history that you have related of the polyester and the A. The polyester foams were used polyether foams. first. They made very good quality rigid foams. Bayer originally announced their polyester based flexible foams in late 1952, I believe, and these systems were received with considerable enthusiasm in Europe, but with very limited (889) enthusiasm in this country. The two main objections in this country from a commercial viewpoint were the degree of softness associated with the foams, the foams were stiffer than customers in this country wanted for comfort cushioning, mattresses and such applications, and also it was judged that in this country the polyester based flexible foams had rather limited resistance to hydrolysis and would not be suitable for a wide range of applications.

"The polyester foams which were developed in a commercial scale in this country after the polyester foams offered three major advantages. One was they had excellent hydrolysis resistance so that that complaint was overcome. Another was that they provided the softness range which customers did want for mattresses, sofa

cushions and so forth.

"The third was that they provided a lower raw material cost for the production of polyurethane foams.

"Q. Was there a particular polyol within the polyethers that was responsible for this? A. I guess the answer to that depends on how specific you want to be in say-

ing a particular polyether. Various suppliers have their own polyethers. Generically, however, the polyethers derived from propylene oxide were the ones that were selected for widest commercial use.

(890) "Q. And is this a propylene oxide based triol? A. Propylene oxide based glycols, specifically di-functional materials, and also triols, and those with higher functionality are used commercially. I don't have production figures available on which one is used most. I believe everybody doesn't have those figures, however, feel sure that it's the triols which are in greatest use.

"Q. And you mentioned, I believe, a billion pound industry; is this in flexible foam? A. No, that's the total polyurethane market. Flexible foam is the major part of it. There are other applications; rigid foams, coatings, adhesives, elastomers, so forth."

(Page 103, line 10-Page 103, line 15)

"Mr. Sweeney: Do you have information, by any chance, of the factor that the flexible foam market would be of this entire market you mentioned?

"A. I think the last published figures in Chemical Engineering News indicated that it was somewhat more than half of the total."

(Page 108, line 2-Page 108, line 25)

- "Q. Earlier yesterday you mentioned trimerization, (891) would you explain what that is? A. In the specific context in which I used the term, I referred to the reaction of three separate isocyanate groups to form a isocyanurate ring. It's a trimerization in the sense that three different isocyanate groups have combined into a single structure.
- "Q. Does this mean that the isocyanate is reacting with itself? A. Yes.
- "Q. And when does this happen? A. It's most likely to happen if there are more free iscovanate groups pres-

ent in a reaction system than will be consumed by other groups more active toward them than the isocvanate groups are reactive toward each other. And it also is promoted by certain catalysts.

"Q. Would one of these other things that the isocvanate

might react with be the polyol? A. Yes.

"Q. In other words, if there wasn't enough polyol to fully react with the isocyanate, you might have trimerization? A. That would be true in a situation where there was not enough of some other compound present such as water or an amine, ves."

- (892)(Page 116, line 20—Page 117, line 14)
- "Q. How closely did you work with Mr. Heiss? Quite closely. We were in the same laboratory. It was a-what was classed as a two-man laboratory, meaning two technical people were in it, two desks, benches around the walls. We had one technician in the laboratory with us a large part of the time. We discussed our interests in polyurethanes more or less daily.

"Q. In your opinion, was Mr. Heiss a good chemist? A. Excellent.

- "Q. Would you be able to see each other's reactions and productions in this laboratory without any difficulty? A. Yes.
- "Q. And these urethane products that Mr. Heiss has developed and which are discussed in his patent, did you see them? A. I saw some of them.
 - "Q. Did you see some of them prepared? A. Yes.
- "Q. Have you prepared urethane products like that vourself? A. Yes."
- Mr. Sweeney: Your Honor, last night Mr. Kel-(904)ton and I discussed the original Heiss notebooks, which are of considerable value. I have had good clear photostatic prints made of the notebooks and

Dr. Herman Francis Mark, for Plaintiff, Direct

we agreed that I could withdraw the originals and substitute these photostats so I don't run the risk of losing them.

(910) HERMAN FRANCIS MARK, called as a witness by the plaintiff in rebuttal, resumed the stand.

Direct Examination by Mr. Kelton:

Q. Dr. Mark, you have been present here in the court-room and heard the testimony? A. Yes.

Q. Do you have an opinion as to whether or not there was an excess of isocyanate in Plaintiff's Exhibit 18, Section B, towards the bottom of the page? By that I mean the reactions employing one or the other of alpha-chloronapthalene and diethylphallate? A. Yes, I have.

Q. Would you like the question reread, Doctor? A.

Yes, I would like to have it reread.

(Question read.)

- Q. What is your opinion? A. My opinion is that there was in excess of TDI employed when the condensate of pentaerythritol and propylene oxide was reacted either in alpha-chloronapthalene solution or in diethylphallate solution with TDI.
- Q. Upon what do you base your opinion, Doctor? A. I have two reasons for this opinion. One is (911) that Dr. Herbst, who carried out these reactions, knew that he was supposed—he had to, he should—apply an excess of diisocyanate.

The other is that the reaction itself, that is, the reaction of TD* with the solution of the condensate in either of the two solvents, offers in fact two independent possibilities to follow the progress of the reaction.

Dr. Herman Francis Mark, for Plaintiff, Direct

Possibilities which can be established reasonably easily. One is that the reaction generates heat and, therefore, as long as it goes on, one feels that there is heat evolved. As soon as the reaction is at the end, there is no further evolution of heat.

The second is that as long as the reaction is not complete, the system is a viscous fluid and as soon as the reaction reaches conclusion, the system gels, so those are two indications as to whether one has reached the end of the reaction.

Dr. Herbst in both experiments, in the small one and in the larger one, carefully added dropwise the TDI and while he added it he established the fact that heat was evolved and he established by shaking it that there was still a viscous liquid present.

So, when he eventually arrived at the end of the (912) reaction in both cases, he had the clear concept or he had actually the observation that now the reaction is at an end because no heat is any more evolved and the material has gelled. As soon as he has reached this stage, he added some more of the diisocyanate.

- Q. Now, Doctor, do you have an opinion as to whether or not there is or I should say was a similar excess of isocyanate in Example III of the Price patent, which is Defendant's Exhibit A? A. Yes.
- Q. If you assume that the example was run in the same way but on a larger scale than the experiment using alpha-chloronapthalene in Plaintiff's Exhibit 18— A. Yes.
- Q. What is your opinion? A. My opinion is the same. An excess was used and the reason is the same.
- Q. In other words, did you mean to indicate that the basis for your opinion is the same? A. Yes.
- Q. As I understand you, Doctor, your reasons are based on, among other things, the manipulative steps that Herbst testified about? A. Yes. They are based on the experimental facts, (913) which were established during his work.

Dr. Herman Francis Mark, for Plaintiff, Cross

Q. Would it be fair to indicate that your reasons were empirical rather than theoretical or what? A. Yes, my conclusions are based on the experimental facts and the procedure and the way he worked under reasons why he felt that the reaction was at an end, an observation of facts.

Mr. Kelton: I have no further questions, your Honor.

Cross Examination by Mr. Sweeney:

Q. Do you still have Plaintiff's Exhibit 18 in front of you, Doctor? A. Yes, I have it here.

- Q. Where in Plaintiff's Exhibit 18 is there any proof at all that Herbst used an excess of isocyanate? A. In Plaintiff's Exhibit 18 there is no proof. The proof is in the description of how he carried out the experiments.
- Q. You testified that one of the things that proved it to you was that the reaction gave off heat? A. That is correct.
- Q. Is there anything about the reaction giving off heat in Plaintiff's Exhibit 18? (914) A. No, but there was a considerable amount of testimony to the fact that this reaction is exothermic.
 - Q. I am talking about Plaintiff's Exhibit 18. A. No.
- Q. There is no indication of how much heat was given off or whether any was given off? A. No.

Mr. Sweeney: I have no further questions, your Honor.

The Court: All right. (Witness excused.)

The Court: Mr. Kelton, anything further?

Mr. Kelton: The plaintiff rests.

Dr. William Colburn, for Defendant, Direct

Mr. Sweeney: Your Honor, may I call Dr. Colburn on surrebuttal on this testimony of Dr. Mark's? That's all, a couple of minutes.

The Court: All right.

(915) WILLIAM COLBURN, called as a witness by the defendant in surrebuttal, resumed the stand.

Direct Examination by Mr. Sweeney:

Q. You have just heard Dr. Mark's testimony on the subject of the excess of TDI.

What is your opinion of Dr. Mark's testimony? A. Well, Dr. Mark gave two reasons why he thought that Herbst's work proved there was an excess of TDI.

One was that heat was developed and that eventually the development of the heat ceased.

Heat is developed in the reaction of TDI with a hydroxyl regardless of the proportions. As soon as the first little bit of TDI is put in, heat is developed and this is shown in Herbst's description of his work, which is Plaintiff's Exhibit 24.

He let it cool and then he added a little more and some more heat was developed and then he cooled that and so on so that right from the very beginning, where there couldn't possibly be an excess, there is heat developed.

The development of heat by itself does not prove that there is an excess.

As the reaction proceeds, the development of (916) heat decreases in rate and that is for two reasons.

One is that the second isocyanate on TDI is less reactive than the first one. So, as soon as we have used up half of the isocyanate in the TDI and we start to react with the second one, the heat development is much diminished.

Dr. William Colburn, for Defendant, Direct

The other part is that for each molecule that reacts there is a certain amount of heat and that is distributed among the mass of material to raise its temperature. It is as if we put a drop of boiling water in a bucket. The whole bucket doesn't reach the boiling point. The quantity of that drop of water and the amount of heat it is introducing into the bucket determines the final temperature.

Here we have an increasing amount of material, as we add the TDI and as it has reacted and cooled under the tap and each additional increment has less effect on the temperature, each additional increment puts in a certain amount of energy, but there is no more for it to be distributed around so the temperature rise decreases as the reaction goes on.

The fact that the rate of heat development slows down doesn't prove that we have ever added enough. That by itself just shows there is a reaction going on, but it (917) is not enough to show when we have added enough of the material or when we have added an excess.

The other point that Dr. Mark made was that the mixture gelled instead of remaining fluid; it became solid.

Now, this reaction without the solvent became hard and brittle. With the solvent, of course, there is plasticizer and so it is not hard and brittle, it is only rubbery or a gel, but it is the same reaction whether the solvent is there or not.

So, you might as well have said that the fact that it got hard and brittle shows that cross-linking took place and the cross-linking is the indication that there was an excess.

If you don't have enough TDI, I agree that you won't get cross-linking and the material won't harden and gel, et cetera, but if we look at Plaintiff's Exhibit 20, a reaction was run where the polyol was the reaction of propylene oxide with sodium, nothing else. Just pro-

Dr. William Colburn, for Defendant, Direct

pylene oxide and propylene oxide has only two hydroxyls in it and the polymer of propylene oxide has only two hydroxyls and this also got hard with no cross-linking possible.

In other words, something in here gets hard (918) when he puts in TDT even if you can't cross-link and, therefore, the fact that it got hard or gelled doesn't show cross-linking took place and, therefore, it doesn't show there was excess material.

Mr. Sweeney: I have no further questions, your Honor.

The Court: Mr. Kelton.

Mr. Kelton: I have no further questions, your Honor.

I think the testimony was unusual and I think it was quite argumentative and I don't choose to cross-examine.

The Court: Thank you, Dr. Colburn.

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